STUDIES ON ASSESSMENT OF SUITABILITY OF TYPICAL SYNTHETIC SURFACTANTS AND ADDITIVE FOR USE IN FOAM CONCRETE PRODUCTION

by

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STUDIES ON ASSESSMENT OF SUITABILITY OF TYPICAL SYNTHETIC SURFACTANTS AND ADDITIVE FOR USE IN FOAM CONCRETE PRODUCTION

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CERTIFICATE

It is certified that the work contained in the thesis entitled **"Studies on Assessment of Suitability of typical Synthetic Surfactants and Additive for use in Foam Concrete Production"** by **Sritam Swapnadarshi Sahu** (Roll No: 146104007), a student of the Department of Civil Engineering, Indian Institute of Technology Guwahati, submitted for the award of the degree of Doctor of Philosophy, has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

Date: 20th March 2019
Place: IIT Guwahati

Dr. G. Indu Siva Ranjani
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Dedicated

To

My Parents

for supporting and encouraging me to believe in myself

My Grandparents

for being my first teachers

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ABSTRACT

Foam concrete which is classified as light weight concrete is basically a cement paste or mortar with air voids entrapped by suitable foaming agent. The key attributes of foam concrete are its low self-weight, high flowability and excellent thermal insulation characteristics. Though it is now widely used in countries like Netherland, Sweden, Germany, USA, Switzerland and UK, however its extensive usage in India is limited by its technical unfamiliarity and non-availability of affordable foaming agents and foam generator.

Review of literature highlighted that although there have been several investigations reported on properties of foam concrete, to date only scarce literature is available on evaluation and improvement of characteristics of foam for use in foam concrete. Hence motivated by the arduous requirement for more studies on surfactant, an in depth study on evaluation of suitability of different synthetic surfactants and additives and its performance in concrete are deemed necessary. The present study is restricted to moist cured foam concrete made of preformed foaming method with the identified surfactants viz. sodium lauryl sulphate (SLS), nonyl phenol ethoxylated (NPE) and additive carboxymethyl cellulose (CMC). The first phase of studies comprises of evaluation of performance of foam produced with one synthetic surfactant SLS and additive CMC adopting two different foam production methods namely mechanical method (stirrer) and compressed air method (foam generator). The relative characteristics of foam and surfactant solution viz., initial foam density, foam stability, foam bubble size and viscosity of surfactant solution produced with synthetic surfactant SLS and viscosity enhancing agent CMC have been studied through a systematic experiment design based on Response Surface Methodology (RSM) for two different foam production methods. Based on comparative studies on foam production methods, it is identified that foam produced based on compressed air method (foam generator) is of better quality with lesser liquid fraction and drainage compared to that of foam produced with stirrer. Hence the further studies are carried out with compressed air method of foam production. As a next step, systematic study on the relative behaviour of foam produced with two synthetic surfactants SLS and NPE and CMC using compressed air method of foam production is carried out. Based on the comparative studies it is observed that NPE surfactant solution is highly viscous with higher initial foam density and relatively lesser early drainage when compared to that of SLS. From the optimization studies, the optimal SLS and CMC concentrations are identified as 5% and 0.2% while the optimum NPE and CMC concentrations are 6.1% and 0.16% respectively. However for both the surfactants the optimum foam generation pressure is fixed as 490 kPa to get uninterrupted foam production.

Having identified their potential basic foaming characteristics as a next step, the suitability of these surfactants and CMC for production of foam concrete need to be evaluated as per ASTM guidelines. ASTM C 796-97 and ASTM C 869 have furnished a way of measuring in the laboratory, the performance of a foaming chemical to be used in producing foam for making cellular cement paste of known design density 641 kg/m^3 and water cement ratio of 0.58. Hence, the suitability of both the surfactants at the optimal foam production parameters chosen is verified with respect to fresh density, compressive strength and water absorption of foam cement paste of density 641 kg/m^3 and the experimental outcomes satisfies the requirements of ASTM C 869 confirming the foam stability. After the assessment of foam performance in cement paste, as a next step, the further evaluation is done for foam concrete for three different target fresh densities 1000, 1500 and 1800 kg/m^3 respectively.

To study the fresh state behavior, the optimum water-solids ratio which would satisfy both the stability and consistence requirement of the mix for different design densities has been determined. Further the relative workability of the mixes with two different surfactants at the identified optimum water-solids ratio is evaluated through measurement of spread in a flow table and flow in modified Marsh cone. For the foam concrete produced with both the surfactants, for a constant water-solids ratio, the spread reduces and flow time increases with an increase in foam volume. Hence, the foam volume has considerable influence on the flow behaviour of foam concrete. Experimental studies have established that the increase in surfactant and CMC concentration which increases the viscosity of surfactant solution also affects the workability related characteristics of concrete. Hence the optimum value of surfactant and CMC concentration need to be checked from point of view of workability and stability of foam concrete mixes also in addition to optimization studies on foam properties. The spreadability requirement to attain the desired design density ranges from 75% to 150% and 45% to 125% for surfactants NPE and SLS respectively.

As the microstructure of concrete has greater impact on mechanical and durability related properties of concrete, the air void system is characterized based on size and shape and further correlated to density and strength of concrete. Initially the effect of surfactant concentration on air void size distribution and circularity is studied. Studies confirms that the increase in surfactant concentration results in reduction in air void diameter and increase in circularity of air voids for foam concrete produced with both the surfactants and this could be attributed to increase in viscosity of surfactant solution. Further the air void size distribution is relatively smaller for foam concrete with NPE when compared to that of SLS. Studies also indicates that the size of larger air voids increases sharply with reduction in density of concrete. Also there is significant reduction in circularity of air voids with reduction in density of concrete. Further it is to be noted that the addition of CMC has resulted in significant improvement in microstructure of concrete with reduced air void sizes for both the surfactants. Supporting the earlier observations, the increase in surfactant concentration and addition of CMC also leads to enhancement in compressive strength and reduction in water absorption and sorption characteristics of foamed concrete due to improvement in microstructure of concrete.

Keywords : Foam concrete, surfactant, density, surfactant concentration, foam stability, viscosity of surfactant solution, response surface methodology, density ratio, water solids ratio, air void parameter, image analysis, compressive strength, water absorption and sorptivity.

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ABBREVIATIONS

LWC	Light weight concrete
LWAC	Light weight aggregate concrete
FC	Foam concrete
SLS	Sodium lauryl sulphate
CMC	Carboxymethyl cellulose
RSM	Response surface methodology
NPE	Nonyl phenol Ethoxylated
WRA	Water reducing admixture
CTAB	Cetyltrimethyl ammonium bromide
SLES	Sodium lauryl ether sulphate
AES	Alcohol ethoxy sulphate
AOS	Alpha olefin sulfonate
CDA	Cocodiethanolamide
SN	Soapnut
PVA	Polyvinyl alcohol
FER	Foam expansion ratio
FGP	Foam generation pressure
SC	Surfactant concentration
GGBS	Ground granulated blast furnace slag
SP	Super plasticizer
CCD	Central composite design
IFD	Initial foam density
FS	Foam stability

NOTATIONS

P	Predicted value
O	Observed value
D	% Difference
w/c	Water cement ratio
D50	Median air void
C50	Median circularity
D90	Bigger size air void
C10	Irregular air void
i	Cumulative volume absorbed per unit area of inflow surface
W_c	Weight of cement
W_s	Weight of sand
W_{TW}	Weight of total water
V_a	Volume of air
V_f	Volume of foam
W_{uf}	Unit weight of foam
W_f	Weight of foam
Φ	Liquid fraction

Chapter 1
INTRODUCTION

1.1 GENERAL

In concrete construction, as self-weight represents a significant proportion of the total load on the structure and hence, attempts to reduce dead load can contribute to major savings in foundation, material, labour, transportation and operating costs. Hence, the light weight concrete (LWC) with density range of 300 to 1850 kg/m^3 (Bing et al. 2012; Narayanan and Ramamurthy 2000; Ramamurthy et al. 2009) is considered as the economical solution for reduction of dead load of the structure. Furthermore, the other added benefits of LWC are better thermal insulation, fire protection and seismic resistance when compared to normal weight concrete. Hence with all the above mentioned advantages, the LWC is destined to become a dominant material for construction in the future.

1.2 LIGHT WEIGHT CONCRETE

Basically common ways by which concrete can be made lighter are by replacing normal weight aggregate with porous aggregate as in light weight aggregate concrete (LWAC) or by introducing air voids in the mortar portion of concrete as called as cellular concrete. Besides, omission of finer sizes of aggregates from aggregate grading can also make the concrete lighter resulting in no fines concrete (Chandra and Berntsson 2002). Out of these, LWAC and cellular concrete are most widely used. The major limitations of LWAC are limited availability of raw material at most locations, production of limited range of densities and requirement of substantial energy for sintering process (Owens 1993). The cellular concrete which has a wide range of density ranging from 300 to 1850 kg/m^3 can be further classified into gas concrete and foam concrete based on the method of pore formation. The cellular structure can be achieved either by adding gas forming chemicals such as aluminium powder or hydrogen peroxide into cement mortar in plastic state (gas concrete) (Kan and Demirboga 2009) or through mechanical means with the help of foaming agents (foam concrete) (Cox 2005).

The main disadvantages of gas concrete are the high energy cost of autoclaving in production and limited to only precast method of production (Aroni et al. 2005; Hamad 2014). Furthermore researchers have reported that the pore structure of concrete is non uniform and precise control of density is difficult in case of gas concrete. Hence foam concrete is more feasible for in-situ applications than gas concrete.

1.3 Foam Concrete

Foam concrete (FC) is either a cement paste or mortar in which the air voids are entrapped in the mortar by suitable foaming agent (Vilches et al. 2012). The cellular structure is produced either by preforming method or mixed foaming method (Bing et al. 2012; Nambiar and Ramamurthy 2007). The preforming method encompasses generating base mix and foam independently and then the foam is completely blended into the base mix. In the mix foaming method, the foaming agent is mixed along with the base mix constituents and during the process of mixing, the foam is produced resulting in cellular structure in concrete (Fig. 1.1). The preformed method is commonly adopted because of much lower foaming agent requirement and precise control over density of concrete.

Foam concrete finds applications in many areas because of its key attributes such as low self weight, high flowability, low aggregate usage, excellent thermal insulation and sound absorbance (Aldridge 2005; Barnes 2009; Kiani et al. 2016; VanDeijk 1919). For instance in a study by Zhihua et al. (2005), it has been reported that the thermal conductivity value of foam concrete varies from 0.16 W/mK to 0.72 W/mK for dry density of concrete ranging from 430 kg/m^3 to 1490 kg/m^3. Further it has been established that foam concrete has 10 times higher rates of absorbing sound compared to dense concrete (Valore 1954b). By proper control in dosage of foam, a wide range of densities of foam concrete can be obtained for application to structural, partition, insulation and filling grades (Bing et al. 2012; Narayanan and Ramamurthy 2000; Ramamurthy et al. 2009). In addition, large amount of industrial waste such as fly ash, ground granulated blast furnace slag (GGBS) can be used in FC production and thus provides a means of economic and safe disposal of waste residues (Kumar et al. 2018; Xin 2016). Also foam concrete blocks can serve as a competitive substitute for bricks which can prevent the depletion of fertile soil needed for agriculture.

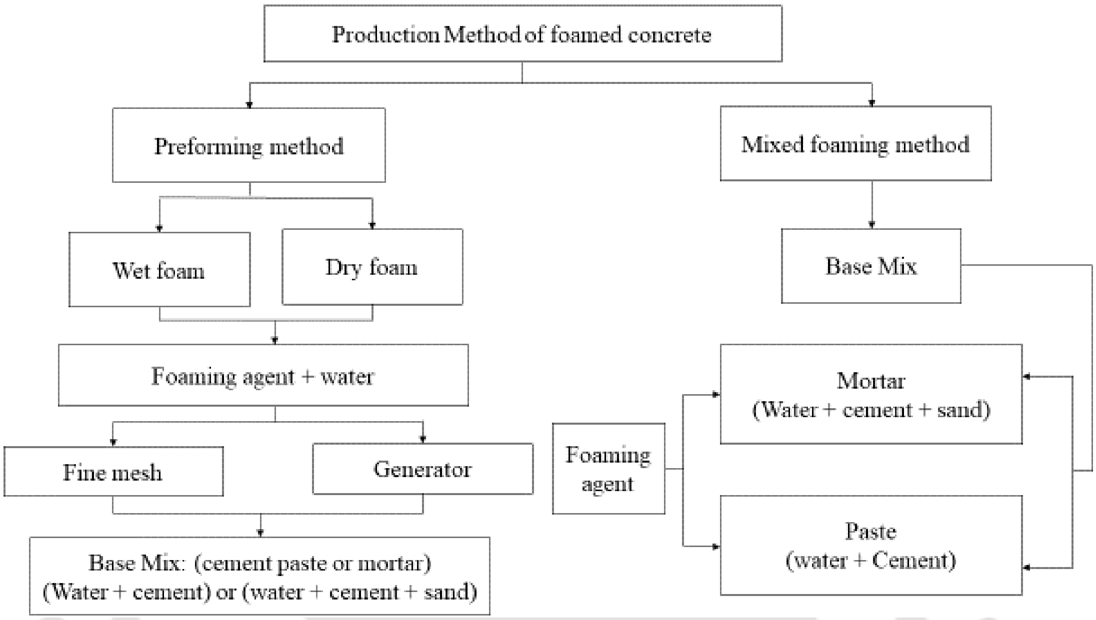


Fig. 1.1 Production methodology of preformed and mixed foamed foam concrete (Hamad 2014)

1.4 ORGANIZATION OF THE THESIS

This thesis is organized into eight chapters. Chapter 1 focuses on the brief introduction of LWC and its classification highlighting mainly on foam concrete and its applications. Chapter 2 provides a critical review on characteristics of surfactant and foam for use in foam concrete production. In addition review on various foam concrete production methods, properties of foam concrete and major research gaps where systematic research is needed. In the same line, Chapter 2 also explains the objectives and scope of present study. Chapter 3 illustrates the relative characteristics of foam and surfactant solution viz., initial foam density, foam stability, foam bubble size and viscosity of surfactant solution produced with synthetic surfactant sodium lauryl sulphate (SLS) and viscosity enhancing agent carboxymethyl cellulose (CMC) thorough a systematic experiment design based on Response Surface Methodology (RSM) for two different foam production methods viz., stirrer (method M1) and foam generator (method M2). Foam generator method based on compressed air method of production of foam is found to result in relatively drier and stable foam suitable for foam concrete production. Hence as a next step, the relative behaviour of foam produced with two different synthetic surfactants sodium lauryl sulphate (SLS) and nonyl phenol ethoxylated (NPE) using compressed air method of production is studied through experimental design based on Response surface methodology as highlighted in Chapter 4. Moreover, the suitability of these surfactants for use in foam concrete production is also checked based on requirements of ASTM C 869.

Having identified their potential as foaming agents, as a next step, the relative workability behaviour of foam concrete produced using these two surfactants SLS and NPE and additive CMC is discussed in Chapter 5. Extensive studies on influence of surfactant concentration, addition of CMC, foam volume and water solids ratio on various fresh state properties of concrete such as fresh density, spread and flow time are studied using the produced foam with two different surfactants as highlighted in Chapter 5. Chapter 6 describes the investigations on influence of type of surfactant, surfactant concentration, addition of additive CMC and foam volume on the microstructure of concrete characterized based on air void size distribution and circularity of air voids. Further the correlation of air void characteristics to compressive strength and density of foam concrete are also investigated. As the water permeation characteristics has important bearing on durability of concrete, water absorption and sorptivity of foam concrete of different densities are also studied as discussed in chapter 6. The typical conclusions drawn based on the studies and the scope for further studies are presented in Chapter 7.

Chapter 2
LITERATURE REVIEW

2.1 GENERAL

Foam concrete finds application in many areas, generally as a function of its relatively lightweight and its beneficial properties in terms of reduction in dead load on structure, excellent thermal insulation and contribution to energy conservation. For production of foam concrete with desired properties, stable and good quality foam is the key requirement. It is to be noted that the selection of surfactant and foam production parameters play a vital role in the properties of foam which in turn affects the properties of foam concrete. However, the literature available on the influence of characteristics of foaming agent and foam on the properties of foam concrete are rather limited. Hence, a more systematic research is needed in this direction. The focus of this chapter is to provide a review on characteristics of surfactant (foaming agent) and foam for use in foam concrete production. In addition, review on various foam concrete production methods, properties of foam concrete and major research gaps where systematic research is needed is also highlighted.

2.2 SURFACTANT

Surfactants are particular type of molecules that contain both hydrophobic and hydrophilic groups. Owing to their unique characteristics, surfactants can self-assemble spontaneously in solutions with various morphologies or enrich at the interface to reduce the interfacial tension (Ghosh 2009). These surfactants show its versatility by finding its application in many industries like foaming in chemical industries, cleaning in automobiles, solubilisation in pharmaceuticals, emulsifying in drilling muds etc. (Porter 1994; Rosen 2004). Furthermore, another important application is as foaming agent for stable foam production in foam concrete industries. Foaming agents are the substances which, when introduced into a liquid, confers on it and possesses ability to produce foam (IS 7597). Not all surfactants can act as a good foaming agent which can produce a stable foam required for production of good quality foam concrete. Hence, selection of surfactant plays a vital role in the properties of foam like foam density, foam output rate, foam capacity, foam stability which in turns affect the properties of foam concrete. In the present context, a comprehensive summary of various properties of surfactant and a classification based on different parameters will be described in detail in sub-sections below.

2.2.1 Properties of Surfactant

The characteristics of surfactant has significant impact on properties of foam as it affects the surface tension and gas liquid interfacial properties. In this section, the focus is given on the important characteristics of surfactant like adsorption, solubility and viscosity as described in the following paragraphs. Table 2.1 summarizes the important characteristics of surfactant and foam studied by various researchers from foam concrete perspective.

2.2.1.1 Adsorption, Solubility and Critical Micellar Concentration

The major characteristics of a surfactant is that it is at a higher concentration at the surface than in the bulk of a liquid. This phenomenon is known as adsorption (Porter 1994). This adsorption at air/liquid interface gives the surface active effects of foaming. The adsorption of a surfactant from solution on to a surface depends on the concentration. Critical micelle concentration of surfactant indicates the concentration at which monolayer adsorption is complete and the surface active properties are at optimum. At concentration above critical micelle concentration, the molecules have no longer any site available for adsorption and hence they form micelles. These micelles have no surface activity since only the monomers are the cause of surface activity. Any further increase in the surfactant concentration above critical micelle concentration does not affect the number of monomers in the solution (Gecol 2006). Hence, there is a considerable practical interest in the critical micelle concentration as it represents in practice, the lowest concentration needed to get maximum benefit in terms of surface activity. Critical micelle concentration depends on the length of the chain of the hydrophobic group. It is reported that critical micelle concentration decreases logarithmically as the number of carbons in the chain of a homologous series increases (Liu et al. 2008). The amount of foam goes through a maximum as the chain length of the hydrophobic group increases (up to a certain extent) but the solubility gets affected (Mehreteab 1999). Shorter the chain length (number of carbon atoms in the chain

(C) < 8), the surfactant is quite soluble in water, but its surfactant properties are minimal. On the other hand, the surfactant with average chain length (C between 10 and 18) is sparingly soluble and has maximum surface active properties. However, when the chain length is very much greater (C above 18) both the solubility and surface active properties are reported to be minimum (Porter 1994). But in practice formulations need good solubility as concentrates need to be diluted. Hence it is to be noted that practical formulae are a compromise between surface active properties and solubility.

2.2.1.2 Viscosity

Viscosity is a property arising from the collision between neighbouring particles in a fluid that moves with different velocities. As the number of particles in a fluid increases (with increase in concentration of surfactant), the viscosity increases (Sharma and Shah 1989). The form of micellar structure varies with concentration, which could be attributed to the variation in viscosity. More commonly the viscosity is defined in terms of surface viscosity and bulk viscosity. Both the parameters are important from point of view of stability of foam as they affect the drainage of foam significantly. The shear velocity of monolayers spread adsorbed either on the surface of liquid or at the interface between the liquids is called as surface viscosity. Researchers have found from their studies that multilayer nature imparts higher viscosity to film than a normal surfactant monolayer (Myers 1999). Also the foaming solution with appreciable viscosity can result in foam with high stability. This is because the drainage rate of foam produced with more viscous surfactant solution is lesser (Pugh 1996).

It is also observed that the chain length of surfactants also affects the viscosity of solution. Straight chain alkyl group is reported to show increased viscosity and inferior solubility compared with similar branched chain surfactants (Porter 1994). Addition of additives like sodium chloride, sodium carbonate and sodium hydroxide can also result in increase in viscosity of solution due to ‘salt effect’ as explained based on Le-Chatelier’s principle (Moore et al. 2010; Siva et al. 2015).

In the same line, Siva et al. (2015) had improved the effectiveness of surfactant sodium lauryl sulphate by adding admixtures like sodium chloride, sodium carbonate and sodium hydroxide. It was reported that the addition of inorganic salts (sodium chloride, sodium sulphate) to diluted surfactant solutions (below about 30%) increased the viscosity, while addition to concentrated solutions decreased the viscosity (Porter 1997).

Table 2.1 Tabulation showing properties of surfactant, foam and the related factors investigated from foam concrete perspective

Sl	Property	Interrelationship and other related factors	Major findings
1	Surface activity	a, c, e, f, g, h, l-o	Studies have proved that at surfactant concentration above critical micelle concentration, no site is available for adsorption and hence there is no improvement in surface activity (Gecol 2006). Amount of foam goes through maximum as the chain length of hydrophobic group increases, but the solubility gets affected (Mehreteab 1999).
2	Viscosity of surfactant Solution	a, c, e, f, h, i, l-o	Viscosity increases with increase in surfactant concentration (Mehreteab 1999). Higher viscous surfactant solution results in foam with high initial density and stability leading to desirable foam concrete properties (Porter 1997; Siva et al. 2015). Viscous nature of protein based foaming solution results in uniform microstructure and enhanced compressive strength of concrete (Brady et al. 2001; Jones 2001; Kim and Jeong 2012; Panesar 2013)
3	Foam stability	a-f, i-k, m-o	Selection of optimum foam production parameters is very important as it affects foam density, texture and ultimately the stability of foam and foam concrete mixes (Ranjani and Ramamurthy 2010).
4	Foam texture	a-f, i-l, n, o	Foam with smaller size bubbles is found to be more stable due to reduction in rate of drainage (Miles et al. 1944). Hence foam with small and spherical bubble is needed to produce foam concrete with uniform and closed cellular microstructure (Brady et al. 2001).
5	Foam density	a-e, i, k-m, o	Foam with high density has thick lamella exhibiting substantially better stability (Amran et al. 2015).
6	Foam expansion ratio	a-e, i, k-o	Foam with very high expansion ratio has minimum lamellar thickness resulting in foam with less stability (Ranjani 2011).

Related factors - (a) Type of surfactant (b) Method of foam production (c) Surfactant concentration (d) Foam generation pressure (e) Use of additives (f) Chain length of surfactant (g) Adsorption of surfactant (h) Solubility of surfactant (i) thickness of foam lamellae (j) Surface activity (k) Viscosity of surfactant solution (l) Foam stability (m) Foam texture (n) Foam density (o) Foam expansion ratio

The authors had observed that foam generated with more viscous surfactant solution has high initial density and stability over time. It is reported that viscosity of solution has linear relationship with foam density and bubble size. The fresh and hardened densities, compressive strength and water absorption of foam concrete produced with high viscous foaming solution were also found to be desirable which showed the effectiveness of the improvement of viscosity of foaming solution.

2.2.2 Classification of Surfactant

Surfactants may be classified in several ways. The simplest way to classify the surfactant is on the basis of its origin and further classification can be done on basis of charges carried by hydrophilic part of surfactant as discussed below.

2.2.2.1 Classification of Surfactant Based on Origin

Fundamentally, surfactants are classified into natural and synthetic type based upon the sources from which they are obtained. The first recognized natural surfactants can be dated back to Egyptian times and were derived by integrating both animal and vegetable oils with alkaline salts (Shehata et al. 2008). Basically, natural surfactants are biological compounds originated from various waste materials, including animal and vegetable sources. Some of the animal protein sources are keratin (hoof and horn meal), cattle hooves and fish scales, casein, blood, bones of cows, pigs and other remainders of animal carcasses (Valore 1954a). There are many plant products which are naturally surface active due to the presence of a compound known as “saponin” and they occur in different parts of a large number of plant species. Some of the saponin rich plant species are soaproot (chlorogalum pomeidianum), soapbark (quillaya saponaria), soapberry (sapindus saponaria), soapnut (sapindus mukurossi) and soapjacob (glinus lotoides) (Oleszek and Hamed 2010; Siva et al. 2017). Saponins have also been reported in grains of many important pulses and leguminous plants which include soya bean, chickpea, mungbean, peanuts, lentils and beans (Singh et al. 2017). They are also present in other edible plant species like oats, leek, garlic, asparagus, tea, spinach, sugar beet, sesame and yam (Oleszek and Hamed 2010). The process of manufacture of natural surfactants is not that simple and involves many stages such as pre-treatment, hydrolysis, neutralization, treatment with preservative, homogenization and stabilization (Lin et al. 2013). Generally pre-treatment involves the use of physical processes like crushing to facilitate size reduction and then steaming (boiling) followed by hydrolysis (Brodeur et al. 2011). Hydrolysis is nothing but breaking down the protein into its simple component amino acids (Pasupuleti and Braun 2010). The process of hydrolysis usually gets improved if the size and crystallinity reduction are carried out prior to it (Brodeur et al. 2011). While there are many means of achieving the hydrolysis, the most commonly adopted methods are prolonged boiling in a strong acid (hydrochloric acid or sulphuric acid) (Pasupuleti and Braun 2010) or strong base (sodium hydroxide, lime or calcium hydroxide) (Lin et al. 2013; Zhang et al. 2014) or using an enzyme (Hill 2010) to simulate the naturally occurring hydrolytic process. If the output of the hydrolysed process is found to be too alkaline or acidic, then the solution derived need to be neutralized. Neutralization is a chemical reaction in which an acid and a base are made to react quantitatively with each other. The pH of the neutralized solution depends on the strength of the reactants.

In general, all the protein foaming agents are a breeding ground for growth of microorganism. Because of the possibility of degradation by bacteria and other microbe (Brady et al. 2001), natural source of protein based foaming agent has to be disinfected with preservative like formaldehyde (Brannan 1995). Then finally the process of homogenization can be carried out with high pressure if the material has to be evenly distributed in order to obtain a homogeneous mass (Dickinson 1997). In order to improve the performance of foam in terms of stability the stabilizers like bone glue (Laukaitis et al. 2005) can be added to the base foaming agent prepared. Despite the complicated manufacturing process there is increasing interest on natural surfactant because of some unique properties like low toxicity and biodegradable nature. In addition to this, it has also achieved its fame in greenhouse effect by reducing CO_2 emission during its manufacture (De et al. 2015). Also its performance when used as foaming agent in concrete has been reported to be relatively better when compared to synthetic surfactant as the protein based foam has uniformly distributed smaller size bubbles (Panesar 2013) with viscous liquid in lamellar layer resulting in more stable foam (Brady et al. 2001; Panesar 2013) which resulted in enhancement of compressive strength of foam concrete (Jones 2001; Kim and Jeong 2012). Besides these advantages, the natural surfactant also has few limitations such as, inconstancy in composition due to variability in raw material, limited storage life (Holmberg 2001) intense stench and its complicated manufacturing process.

To overcome these limitations of natural type, synthetic foaming agents are introduced which is basically man-made chemicals that can easily dissolve in water and produces air bubbles. Table 2.2 presents an overview of various surfactants which have potential for use in foam concrete production as reported in literature. Synthetic type foaming agent has greater degree of application because of its high productivity of manufacturing process, strict control over its composition and longer storage life (Ranjani and Ramamurthy 2010). However the major limitation of synthetic foaming agent is the problem of compatibility with other admixtures which are used in concrete. Particularly, the self-flowing nature of foam concrete necessitates the use of water reducing admixture (WRA) for reduction in water demand. However, the dosage of WRA have to be carefully studied with respect to concrete, as it would affect the stability of foam produced with synthetic surfactants (Jones and McCarthy 2006).

2.2.2.2 Classification of Synthetic Surfactant Based on Charges

In general the hydrophobic part of surfactant consists of a long chain hydrocarbon group whereas the hydrophilic head which can be ionic or non-ionic is a highly polar group (Myers 2006). On the basis of presence or absence of charges in the polar group of surfactants, they are classified as ionic or non-ionic as discussed below.

Ionic Surfactant

Surfactant made up of molecules that have either negatively or positively charged heads are generally termed as ionic surfactant. sodium lauryl sulphate (SLS) and cetyltrimethyl ammonium bromide (CTAB) are commonly used ionic surfactants in foaming process (Amaral et al. 2008; Bera et al. 2013; Ospanova et al. 2014). Ionic surfactant can contribute to foam formation and stabilization because of electrical double layer present at interface which interact with opposing interface to form disjoining pressure and thus results in more stable foam (Myers 1997). These ionic surfactants are further classified into cationic and anionic on basis of specific charge heads and their properties vary depending on its charges.

Cationic Surfactant

Cationic surfactants are generally positively charged head group which can be broadly categorised into long chain amines and quaternary ammonium salts (Malik et al. 2011). Mostly cationic surfactants are water soluble when there is only one long hydrophobe.

Table 2.2 An overview of potential foaming agents for use in foam concrete production

Foaming agent	Classification of foaming agent	Properties Studied		Major Findings
		Foam	Foam concrete	
Sodium lauryl sulphate (SLS)	Anionic Surfactant	i - v, vii	viii – xii, xiv - xviii, xx	Addition of sodium admixtures namely NaCl, Na_2CO_3 and NaOH is reported to improve the foam properties such as initial foam density and stability and subsequently enhanced the performance in cement based mixes (Siva et al. 2015). Use of fly ash as cement replacement material improves the microstructure and the mechanical properties of foam concrete and hence can be extended for use in structural applications (Karthikeyan et al. 2015)
Sodium lauryl ether sulphate (SLES)	Anionic Surfactant	i - v	viii – xii, xiv - xviii	Requirements of ASTM C 869 for foam cement paste are fulfilled when the optimum foam production parameters are adopted (Ranjani and Ramamurthy 2010). Studies on durability related properties proved that the cell like structure of foam concrete do not necessarily make foam concrete less resistant to penetration of aggressive ions (Ranjani and Ramamurthy 2012).
Alcohol ethoxy sulphate (AES)	Anionic Surfactant	i, iv, vi, vii	viii - x, xii, xiii, xv	Thermal conductivity of foam concrete with AES is reported to range from 0.12 to 0.421 W/mK for dry density of concrete ranging from 440-1500 kg/m^3 (Kim and Jeong 2012).
Alpha olefin sulfonate (AOS)	Anionic Surfactant	i, iv, vi, vii	viii - x, xii, xiii, xv	Concrete mixes produced with AOS Foaming agent has higher workability which can be attributed to the lower surface tension of foaming agent solution (Kim and Jeong 2012). Also it is recommended for use in partial load bearing insulation elements because of its higher compressive strength.

Table 2.2 An overview of potential foaming agents for use in foam concrete production (Continued…)

Sodium oleate	Anionic Surfactant	ii	viii, xiv	Sodium oleate when used in cement based mixes tends to show slightly low initial foam volume because of inhibiting effect of calcium ions released by cement but however the stability of foam remains unaffected (Corr et al. 2002).
Sulphanol	Anionic Surfactant	i - v	viii - xi, xiii, xiv	Corr et al. (2002) observed that the foaming ability of sulphanol is relatively less affected by the calcium ions in cement paste (Corr et al. 2002).
Cetyltrimethyl ammonium bromide (CTAB)	Cationic Surfactant	vi	viii - xi, xiii, xiv	The morphology of pore structure changed from monodispersed to highly connected structure when the density of foam concrete was varied from 551- 254 kg/m^3 (Samson et al. 2017).
Cetrimide	Cationic Surfactant	vi	viii - xi, xiii, xiv	Suitable for mix foaming method, as a significant reduction of 50% in density of concrete was realized with small increase of 0.2% dosage of cetrimide (Samson et al. 2017).
Neopor	Aqueous protein hydrolysate	vi	viii - xi, xiii, xiv	Samson et al. (2017) observed from his comparative study that a decrease in dry density of concrete by 100 kg/m^3 resulted in reduction in thermal conductivity of 0.033W/mK and hence foam concrete made with neopor surfactant is found to have relatively low thermal conductivity (Samson et al. 2017).
Microair	PEG fatty acid	vi	viii - xi, xiii, xiv	The surface tension of foaming agent solution prepared with microair is reported to as low as 25 mNN/m and hence it is expected to result in more stable foam leading to improved mechanical performance of foam concrete (Samson et al. 2017).
Cocodiethanol amide (CDA)	Non-ionic Surfactant	i - v	viii - xi	In a comparative study of CDA with ionic surfactants, though the foam produced with CDA had good stability, but the output rate was low due to its high viscous nature (Ranjani and Ramamurthy 2010).
Vegetable Soap	Natural Surfactant	i, iv, vi, vii	viii - x, xii, xiii, xv	The quantity of vegetable oil needed for foam production is less as its foam density is as low as 33 kg/m^3 (Kim and Jeong 2012).

Table 2.2 An overview of potential foaming agents for use in foam concrete production (Continued…)

Fe-Protein	Natural Surfactant	i, iv, vi, vii	viii-x, xii, xiii, xv	In a comparative study with synthetic surfactants, the compressive and flexural strength of foam concrete produced with Fe-Protein is reported to be relatively higher and this can be attributed to the stable nature of foam produced with thick lamellae and high density (Kim and Jeong 2012).
Soapnut (SN)	Plant based	i, ii, v	viii - x, xiv, xv, xx	Surfactant solution prepared by water soaking of SN pericap and then further heating to 80^{O}C resulted in relatively better foam density when compared to simple method of just water soaking of SN pericap without heating. Also the foam produced with SN when used in foam cement paste is reported to result in considerable delay in the setting time (Siva et al. 2017).
Animal hoof and horn	Animal protein based	ii, iv	viii, x, xiii	Foam concrete prepared with animal hoof and horn protein surfactant is reported to possess well pouring stability (Lin et al. 2013).
Baijiu vinasse	Plant protein based	ii, iv	-	The properties of foam such as foamability and foam stability as determined by Ross-Miles meter is found to be satisfactory proving the huge potential for application of Baijiv vinnase as surfactant (Zhang et al. 2014).

Surfactant and foam related properties -

(i) Foam density (ii) Foam stability (iii) Foam output rate (iv) Foam capacity (v) Foam texture (vi) Surface tension of surfactant solution (vii) Viscosity of surfactant

Foam concrete properties - (viii) Fresh density (ix) Dry density (x) Compressive strength (xi) Water absorption (xii) Flexural strength (xiii) Thermal conductivity Microstructure (xv) Workability (xvi) Sorptivity (xvii) Shrinkage (xviii) Sulphate resistance (xix) Chloride resistance (xx) Setting behaviour

However, the product becomes sparingly disperse in water and soluble in organic solvents when there are two or more long chain hydrophobes (Tadros 2014). In general adsorption and surface active properties are reported to be higher for cationic surfactants with lower solubility. Many cationic surfactant possesses good foamability and stability but due to its high cost it counts relatively less use in industry (Ghosh 2009). Cetyltrimethylammonium bromide or CTAB (Quaternary ammonium surfactant), a cationic surfactant have been used in foam concrete production by few researchers (Qin et al. 2014; Samson et al. 2017). These salts are formed by the neutralisation of amine with an acid in aqueous solution. These salts of fatty amines with carbon chain length 12-16 are most effective and proved successful with many acids like acetic acid, glycolic acid and lactic acid (Porter 1994).

Samson et al. (2017) compared the performance of two cationic surfactants namely CTAB and Cetrimide with respect to fresh and hardened state properties of foam concrete. Cetrimide and CTAB were reported to be more suitable for mix foaming method, as a significant reduction of 50% in density of concrete was realized with a small increase in dosage of 0.2% above the base dosage of 0.05% of the above surfactants. The structure of pore system was also found to vary with the density of concrete. The authors had reported that when the density of foam concrete made with CTAB surfactant was varied from 551-254 kg/m^3, the porous structure changed its morphology from monodisperse structure to a highly connected structure. The greater amount of foam in mixes with lower density could be attributed to highly connected structure formed as a result of merging of air voids (Nambiar and Ramamurthy 2007). For the same density, foam concrete with cetrimide is reported to perform better in terms of compressive strength and thermal conductivity when compared to CTAB.

Similarly, another class of cationics are long chain amines, which are positively charged with pH less than 7. However, at high pH (> 7) the long chain amines become no longer charged and lose their activity. On the other hand the quaternary ammonium salts remain charged and retain their activity at all pH conditions (Zhang et al. 2003).

Anionic Surfactant

Anionic surfactants are generally negatively charged head group which can be carboxylate, sulfate, sulfonates and phosphate groups (Kronberg et al. 2014). Ease and low cost associated with its manufacturing process make the anionic surfactant so popular for various applications like detergency, personal care products, emulsifiers and soaps

(Yokoi et al. 2004). The cations most commonly used are sodium, potassium, ammonium, calcium and various amines. The significant features of few important groups of anionic surfactant which are most commonly used in foam concrete production are discussed below.

One of the main types of anionic surfactants commercially available are the carboxylate soaps. Soaps in general are the alkali metal salts of the carboxylic acids of fatty acids. Few of the carboxylate soaps which are commonly used in foam concrete production are sodium lauryl sulphate (SathyaNarayanan and Ramamurthy 2012), sodium oleate (Corr et al. 2002), alpha olefin sulphonate (Kim and Jeong 2012) and sulphanol (Ranjani and Ramamurthy 2010). As mentioned earlier, the solubility of a surfactant depends on the chain length of the hydrophobic group. Hence, the anionic surfactant made from C12 fatty acid like sodium laurate is soluble in water. But surfactants like sodium oleate made from C18 fatty acid is very slow to dissolve. Hence, it can be generalized that below C8, the products are very soluble in water, between C8 and C18 sparingly soluble and above C20 insoluble in water. It is to be noted that optimum surface active properties are obtained with the sparingly soluble products (Porter 1994). Any structural modification that leads to lowering the critical micelle concentration of surfactant by increasing the chain length of anionic surfactant can be expected to increase its efficiency as foaming agent. This is because longer chain length may greatly expand the surface area which can attribute to more rapid reduction in surface tension and hence, results in enhancement of efficiency of foaming agent (Myers 2006). Among the soaps made from fatty acids, maximum foam production is reported from surfactants like sodium laurate and sodium myristate made from C12-14 fatty acids. Sodium stearate gives rich creamy foam, but oleates, laurates and tallates give more open foam (Porter 1994).

The next important class of anionic surfactants are the sulphates. The commercial products of sulphates used as surfactants in foam concrete production are fatty alcohol sulphates (e.g. sodium lauryl sulphate), ether sulphates (e.g. sodium lauryl ether sulphate), nonyl phenol ether sulphates and fatty acid alkanoamide ether sulphate. Most surfactants of sulphate group show salt effect when inorganic additives are added as discussed earlier. Among the sulphates sodium lauryl sulphate (SLS) is more commonly used (Azira et al. 2008; Bera et al. 2013) and found to produce desirable results in terms of stable foam concrete mix when used in foam concrete production as reported by many researchers

(Karthikeyan et al. 2015; Ranjani and Ramamurthy 2010; Siva et al. 2015). Ether sulphates is reported to perform better compared to alcohol sulphates and this is attributed to the presence of the polyethylene oxide which confer improved solubility on ether sulphates (Porter 1994). Surfactant mixture of 2% sulfanol (sodium dodecyl benzene sulphonate) as foaming agent and 0.3% bone glue hydro solution as stabilizer in the ratio 1:0.15 is reported to produce stable foam for use in foam concrete production (Laukaitis et al. 2005).

Zwitterionic surfactant

Zwitterionic surfactant commonly called as amphoteric or ampholyte contains both the charges positive and negative on the surface active part of the molecule. High stability foam with less irritation to skin are the significant advantages of zwitterionic surfactant (Effendy and Maibach 1996; Seredyuk et al. 2001). Cocoamido-propyl betaine is commonly used zwitterionic surfactant (Staszak et al. 2015). As the surfactant carries both the charges, it is found to be compatible with both cationic and anionic surfactants by choosing the right conditions when used in mixed surfactant system (Knepper and Berna 2003). Zwitterionic surfactants are mostly compatible with all class of surfactant except at low pH with anionics, where they give a precipitate. Hence, best foaming properties is obtained at alkaline pH. Zwitterionic surfactant shows excellent foam stability when added to ether sulphate solutions, particularly sulphobetaines. Though these surfactants have absolute potential (Porter 1994) but not many researches have been reported on its use in foam concrete production.

Non-Ionic Surfactant

These surfactants are second most widely used in the industries like textile, food, paper, plastic etc. Most commonly used non-ionic surfactants are Triton X-100 (Wei and Liu 2000), Tween 80 (Amaral et al. 2008) and cocodiethanolamide (Ranjani and Ramamurthy 2010). The size of micelle for nonionics is very much larger than that of anionics or cationics. The shape and size of a micelle are a function of the molecular geometry of its surfactant molecules which generally depends on structure of surfactant and also environmental conditions such as surfactant concentration, temperature, pH etc. (Ghosh 2009). Basically non-ionic surfactants are reported to possess lesser critical micelle concentration value than ionic surfactants because the electrostatic repulsion of the head groups in the periphery of ionic micelles makes micellization more difficult (Liu et al. 2008). Also, when inorganic salts like sodium chloride, sodium bromide and magnesium chloride are incorporated to non-ionic surfactant, it results in further reduction of critical micelle concentration and this reduction is more significant when used in mixed surfactant system with the anionics than with cationics. In addition to that, temperature has also significant effect on surfactant properties. With increase in temperature, critical micelle concentration reduces and the molecule becomes more insoluble and thus becomes more surface active (Porter 1994; Schramm and Marangoni 2000). Besides the above mentioned, as the non-ionic surfactants do not have any significant electrical charge on their surface active part, there is very little or no electrical interaction between the head groups resulting in foam with lesser stability when compared to ionic surfactant (Ghosh 2009).

Composite foaming agent

With the continuous search for improving surfactant properties, mixed surfactants or composite foaming agents have recently emerged to be the one that exhibits interesting synergistic interactions. Several combinations of cationic, anionic and non-ionic surfactants were adopted by various researchers for their studies (Amaral et al. 2008; Lunkenheimer and Malysa 2003; Wang et al. 2014)

Composite foaming agent made with combinations of different types of surfactant is reported to produce better quality foam than the individual components (Khimani and Vora 2011). It can be further postulated that the tendency to form aggregated structures (mixed micelle) in solutions containing mixture of surfactants would be substantially different from solution involving only the pure surfactant (Harwell and Scamehorn 1992).

Ospanova et al. (2014) have reported based on their studies that composite foaming agent made of combination of sodium lauryl sulphate (SLS) and polyvinyl alcohol (PVA) and another composite combination of cetyltrimethylammonium bromide (CTAB) and polyvinyl alcohol performed better in terms of stability and foaming property than their individual components (SLS or CTAB). This is because, the presence of PVA resulted in increased minimum equilibrium film thickness of film around bubble and hence resulted in enhanced foam stability. In addition to above, in presence of PVA, the critical micelle concentration of surfactant is found to decrease which economizes the requirement of surfactant for stable foam production. Also while choosing the combinations of composite foaming gents, the following guidelines can be considered. Anionic surfactants are generally not compatible with cationics and vice versa. On the other hand most of the non-ionic and true amphoteric are compatible with each other and with anionic or cationic (Bera et al. 2013).

2.3 Foam

A mass of gas cells separated by thin films of liquid and formed by the juxtaposition of bubbles, giving a gas dispersed in a liquid is called foam. The properties of foam are greatly influenced by the characteristics of foam, surfactant and method of production of foam as discussed in the following sections.

2.3.1 Methods of Production

It is a well-known fact that pure liquids cannot foam and the presence of surfactant is essential for making and stabilising a foam (Birdi 2009; Pugh 1996). Most of the foam production systems include surfactant solutions, expansion gas (air) and an equipment to combine, mix and deliver the foam product (Aubert et al. 1986). One traditional and most familiar way of preparing foam is by using a dispersion technique, which consists of mechanical shaking of surfactant solution or whipping a surfactant solution mechanically with a high speed stirrer or fan (Krzan 2013). Researchers (Gido et al. 1989; Hirt et al. 1987) have proved that the mean bubble size of foam produced decreased with increase in rotational speed of stirrer. Hence, the speed of mixing is an important parameter for achieving good quality foam with this approach. However, this method is not particularly satisfactory since the accurate control of the amount of air which is incorporated in the liquid is difficult to achieve.

The other alternate method is aerosol method where the liquid is supersaturated with gas by dissolving the gas into the surfactant solution under pressure and then releasing the pressure as done in pop drinks (Walstra 1989). The high cost associated with this method limits its wider application for foam generation (Aubert et al. 1986). The most widely used method of foam production for use in concrete is compressed air method wherein the foam is generated by mixing compressed air and surfactant solution in high density restrictions. Studies have proved that the foam produced by compressed air method is relatively more stable as the bubble size distribution in foam is narrower (Magrabi et al. 1999).

2.3.2 Classification of Foam

One of the most common schemes of classification is based on its degree of stability. They may also be classified on the basis of its liquid fraction as discussed below.

2.3.2.1 Based on the Liquid Fraction

Many of the physical properties of a foam depend on its liquid fraction i.e the volume ratio of liquid and gas (ϕ). Based on the liquid fraction, the foams are classified into wet and dry foam as discussed below. Wet foam usually refers to foam with liquid fraction higher than 0.15 (Hutzler et al. 2005). Lykelama calls system with ϕ greater than 0.37 as extremely wet foam (Bergeron and Walstra 2005). Foam produced based on dispersion technique and aerosol method can be classified as wet foam as the liquid fraction is higher. As suggested by Aldridge (2005) wet foam can also be produced by spraying a solution of foaming agent over a fine mesh. The foam produced by above mentioned method has large loose bubble structure with 2-5 mm bubble size and is relatively less stable (Barnes 2009). Hence, such foam is not suitable to produce foam concrete with uniform microstructure. As the gas fraction in foam becomes high enough, bubbles are forced into contact and form polyhedral structures leading to dry foam (Bergeron and Walstra 2005). Foam with ϕ less than 0.05 is considered as extremely dry foam (Hutzler et al. 2005). Foam produced using compressed air method can be classified as dry foam in which the foam is produced by forcing diluted solution with compressed air through a series of high density restrictions in a chamber. Generally, dry foam is relatively stable and is recommended for foam concrete production.

2.3.2.2 Based on Degree of Stability

It is extremely important to have a foam, which can persist for a considerable time till the concrete sets and attains a uniform cellular structure which is an important requirement for cellular concrete. So, based on stability point of view, foam can be generalized into unstable, metastable and solid foams. Unstable foams are low persistence foams or transient foam and as their name implies they remain for a very short time and collapse as a result of the overwhelming effects of surface tension and gravitational forces. The lifetime of these foams ranges from several seconds to about 20 seconds (Lunkenheimer and Malysa 2003). Metastable foam possess a persistence from few minutes to months (Myers 2006). They can withstand ordinary disturbances (Brownian fluctuations) but collapse due to abnormal disturbance like evaporation or temperature gradient (Kontogeorgis and Kiil 2016). They are stabilized by the presence of surface active materials at the liquid/gas interface which can retard the loss of drainage of liquid from the area between bubbles.

Solid foams could be considered as thermodynamically stable since they possess a mechanically rigid structure formed as a result of irreversible chemical process during or just after foam formation. Most solid foams are made using blowing agent (which supply the gas) by boiling, upon increase of temperature or release of pressure, or by chemical decomposition. Flurocarbons (currently prohibited in many uses), CO_2 and N_2 are commonly used as final products of such blowing agent.

2.3.3 Properties of Foam

In this section the important properties of foam viz. foam texture, density and stability are reviewed to provide information on its impact on foam concrete properties.

2.3.3.1 Foam Texture

Foam texture refers to shape and size of bubbles in foam (Bonsu et al. 2016). The shape of bubbles in foam in general can be broadly classified into spherical and polyhedral (Vijayaraghavan et al. 2009). A variety of techniques have been used in earlier studies to study the bubble size distribution: the conductivity probe, the optical fibre based method, light transmission procedures and photographic methods to mention a few. Among the above mentioned methods, photographic methods are reported to be non-intrusive as it does not change or distort the bubble size distribution (Magrabi et al. 1999). The bubble size in preformed foam added to concrete is reported to range from 0.1 to 1mm (Wei et al. 2013). As highlighted in numerous studies there is a strong relationship between bubble size distribution and rate of drainage (Magrabi et al. 1999). The foam with smaller size bubbles are reported to be more stable due to reduction in rate of drainage (Miles et al. 1944). In addition, more uniform bubble size distribution also enhances foam stability (Magrabi et al. 2002; Sarma and Khilar 1988; Sheng 2013). It is clear from the above observation that the foam with fine texture (small spherical bubbles) is needed to produce foam concrete with uniform and closed cellular microstructure (Brady et al. 2001).

2.3.3.2 Foam Density

Foam density refers to unit weight of foam. This parameter is useful to calculate the volume of foam which is the governing parameter for achieving a desired density of foam concrete (Ranjani and Ramamurthy 2010). For foam concrete, (ASTM C ASTM 796) has recommended a foam unit weight range of 32-64 kg/m^3 with a remark that this range could be adjusted according to manufacturer’s recommendation based on foam chemical and generator used.

Also it is important to note that the foam density is dependent on various factors like type of surfactant (Hamad 2014), surfactant concentration (Ranjani and Ramamurthy 2010), additives (Colak 2000), viscosity of foaming solution (Kroezen et al. 1988; Siva et al. 2015), thickness of lamellae (Krzan 2013) and foam generation pressure (Ranjani and Ramamurthy 2010). The influence of foam production parameters namely surfactant concentration and foam generation pressure on foam density shall be discussed in later section.

Generally, the bubbles in the foam are surrounded by the layer of liquid called as lamellae. The thickness of the lamellae and the properties of liquid in the lamellae influences the properties of foam to a greater extent. Researchers have reported that, a critical thickness of 5 to 15 nm is required to support the pressure in the bubbles (Myers 1997). Most commonly, the foam with high density has thicker lamellar layer than foam with low density. Hence, when the thickness of lamellar film around the bubble is greater than the critical thickness required, the foam stability is reported to be good. Few researchers have concluded that synthetic foam tend to have greater expansion and hence lower density when compared to protein based foaming agent (Amran et al. 2015). So, in order to use the low density foams for foam concrete production, it would be advisable to add some foam thickening agent as additives to improve the density of foam. Various researchers have incorporated different additives like carboxyl methyl cellulose (Colak 2000), sodium hydroxide, sodium carbonate and sodium chloride (Siva et al. 2015) to enhance the viscosity of foaming solution which resulted in improvement of foam density and subsequent improvement in foam concrete characteristics

2.3.3.3 Foam Stability

The ability of foam to withstand spontaneous collapse or breakdown from external causes is called foam stability. The foam stability reflects the life of lamellae in the generated foam (Ranjani and Ramamurthy 2010). It is the function of various parameters like thickness of lamellae (Callaghan 1989), viscosity of liquid (Brown et al. 1953), elasticity of film (Murray and Ettelaie 2004) and bubble size (Wilde 1996). Furthermore, foam stability is an essential parameter for foam concrete production to ensure a uniform cellular structure of concrete as the bubble should remain stable till the concrete sets. There are various mechanisms affecting the stability of foam like gravitational drainage from the films, coarsening caused by inter-bubble gas transport, coalescence of adjacent bubbles due to rupture of inter-bubble lamellae etc. (Murray and Ettelaie 2004; Tan et al. 2005). Coarsening which is the inter-bubble diffusion causing the expansion of larger bubbles at the expense of the smaller bubbles is a difficult phenomenon to measure (Magrabi et al. 2001). Hence, the drainage rate is often used to characterize the stability of foam which can be measured either as free drainage or forced drainage (Hutzler et al. 2005a). Generally, freshly made foam starts draining due to gravity and this naturally occurring process is called free drainage. The main problem states when to start the experiment, since foam starts draining as soon as it is formed (Hutzler et al. 2005).

On the other hand, forced drainage is also difficult to manage, as the high pressure may lead to breakdown of bubbles. Foam stability have also been assessed through studies on the variation of height of foam (Lee et al. 2014; Lunkenheimer and Malysa 2003; Sanova and Lisitsyn 2012) or volume of foam (Shrestha et al. 2006; Weschayanwiwat et al. 2005) or density of foam with time (Ranjani and Ramamurthy 2010). ASTM C 796 and (ASTM C 869) has prescribed standard guideline to evaluate the performance of foaming agent for use in preformed foam concrete which has been taken as a measure of stability of foam in concrete by various researchers (Ranjani and Ramamurthy 2010; Siva et al. 2015). Foam concrete has to meet the requirements of ASTM C 869 with respect to fresh density, oven dry density, compressive strength, split tensile strength and water absorption for its suitability in foam concrete production.

Various studies have concluded that stability of foam could be enhanced through addition of additives like bone glue (Laukaitis et al. 2005) and viscosity enhancing agent (Siva et al. 2015). Use of the fine fillers like fly ash and pulverized sand in concrete is also reported to enhance the foam stability as the fine material forms a uniform coating around the lamellae and prevent the coarsening effect (Awang et al. 2012; Nambiar and Ramamurthy 2006; Nambiar and Ramamurthy 2008).

2.3.3.4 Foam Expansion Ratio

Foam expansion ratio (FER) is a measure of foam capacity or foamability of foaming solution (Vananuvat and Kinsella 1975). The ratio of volume of foam formed to the volume of foaming solution used to generate the foam is called foam expansion ratio (Magrabi et al. 2002). Commonly speaking, expansion ratio or foam multiplicity or foam capacity are user friendly term for foam users since it gives a volume for measurement of foam that could be produced from unit quantity of foaming solution (Ospanova et al. 2014;

Sanova and Lisitsyn 2012). Few researchers (Boos et al. 2013; Marinova et al. 2009; Varade et al. 2011) have used technique like foam scan apparatus to measure the foamability wherein the ratio of volume of foam to volume of gas introduced for formation of foam is measured as foamability. FER depends on properties of surfactant and foam production parameters namely surfactant concentration and foam generation pressure. The foam to be used in preform foam concrete production should have FER in the range of (1:15.6 to 1:31.25) as derived from foam density range prescribed by ASTM C 796. Foam with very high foam expansion ratio say greater than (1:50) has very thin lamellar layer resulting in foam with less stability as discussed in earlier sections. Such kind of over expanded foams of lower foam density are likely to collapse and increase the concrete density (Ranjani 2011). On the other hand, foam with less expansion ratio (less than 1:15) has less amount of air resulting in poor quality foam.

2.4 Foam Concrete Production Parameters

On the basis of the production methodology, foam concrete is classified into preform or mix foam foam concrete (Richard and Ramli 2011). In preform method of foam concrete production, the foam generated using any one of the methods as discussed in earlier section 2.3.1. is added to the base mix (mortar without foam) and mixed thoroughly till a homogeneous mix is obtained. Similarly mix foam foam concrete is produced by mixing all the ingredients including the surfactant, cement and water in the mixer machine at a high speed. During the process of mixing, the foam is generated which on setting leaves a cellular structure (Bing et al. 2012). Hence, the mix foam method does not require a separate foam production equipment like foam generator and its efficiency is governed by type of surfactant, its dosage, mixing speed, duration of mixing and type of mixer machine. On the other hand, the efficiency of preformed method depends on foam generation pressure also in addition to the parameters mentioned above. The influence of the various foam concrete production parameters on the quality of foam concrete will be discussed in the following sections.

2.4.1 Foam Production Parameters

2.4.1.1 Foam Generation Pressure

The pressure at which foam is generated from foam generator is referred as foam generation pressure (FGP). This is one of the production parameters which controls the mixing of air with foaming liquid and hence governs the quality of foam in preformed method of foam concrete production using foam generator. For a given surfactant concentration, the foam density is observed to be in direct relation with FGP. The foam generation pressure also determines the size of bubble in foam. Smaller the size of bubble in foam, more will be the stability of foam and uniform will be the microstructure of concrete. At low pressure (< 30 kPa) the physical properties of solutions, density, viscosity and dynamic surface tension determine size of bubble being formed. However, as the pressure and hence the flow rate of air increases, the solution effects are neglected and the bubble diameter is determined by the generation pressure (Kearsley and Visagie 1999; Nambiar and Ramamurthy 2007; Quebaud et al. 1998; Wilde 1996). Adoption of very lower FGP resulted in poor quality of foam due to inability of compressed air to penetrate into the foam solution. This in turn resulted in poor cellular structure when such foam is used in foam concrete. On the other hand, at very higher FGP, the foaming solution also comes out along with foam due to turbulence in the container resulting in foam with higher density and poor stability which was confirmed by higher drop in density with time (Ranjani and Ramamurthy 2010; Siva et al. 2015). Subsequently this resulted in poor quality of foam with lesser amount of air leading to failure in the achievement of design density of foam concrete. Similar observations were reported by researchers Siva et al. (2015) stating that it is difficult to generate good quality foam with generation pressure below 78 kPa and above 137 kPa. Therefore, selection of optimum FGP is very important as it affects the foam density, bubble size and ultimately the stability of foam and foam concrete mixes. Also it is to be noted that the optimum foam generation pressure has to be derived based on designed experimental trials only.

2.4.1.2 Surfactant Concentration

Surfactant concentration (SC) represents the concentration of surfactant in the foaming premix solution prepared. It can also be expressed in terms of dilution ratio, say 1:x which means one part of foaming agent is diluted with x parts of water. Studies have been conducted by few researchers on the effect of variation of SC on the foam properties and it has been proved that adoption of very low SC resulted in foam with very high foam density but the stability of foam thus produced was found to be very poor which was confirmed by higher rate of drainage (Ranjani and Ramamurthy 2010). In this line, Siva et al. (2015) stated that when the adopted surfactant (sodium lauryl sulphate) concentration was below 0.5% foam could not be produced as it is mostly water with lower concentration of surfactant. But dosages beyond 2.5% resulted in no significant variation in density of foam.

In general the usage of higher SC resulted in more viscous and stable foam. However, the usage of higher SC is not economical for foam concrete production. Hence, the selection of optimal SC to produce stable foam is deemed necessary for economical foam concrete production. To simplify these optimization studies on SC, critical micelle concentration of surfactant can be used as a guiding parameter to derive the optimum surfactant concentration. In addition to SC and FGP, mixing speed and the type of mixer machine also greatly influenced foam concrete production which will be discussed in the next section.

2.4.2 Mixing Speed, Mixer Machine, Batching Sequence

Most common types of mixers such as tilt drum or pan mixer which is commonly used for concrete or mortar can be used for foam concrete (Karl and Worner 1994). The type of mixer, batching and mixing sequences of foam concrete depends upon whether it is preform method or mix foam method of production. For production of preform foam concrete the following batching sequence was reported in earlier studies. The mixing sequence constitutes first combining of cement and filler with water (including superplasticizer) for 1 minute with a trowel to minimise the material loss that can occur from mixing in an open mixer. Then using the mixer, the materials were mixed for 4 minutes until a homogeneous base mix was achieved. To the base mix thus prepared, the necessary volume of preform foam is introduced and thereafter mixed for at least 2 or 3 minutes in order to get uniform distribution of foam (Jones 2001; Nambiar and Ramamurthy 2008). On the other hand, in mix foam method, the foaming agent is mixed along with base mix ingredients and after a short period of slow mixing, the mixer was operated at 80 to 90 rpm for a period of 2 to 3 minutes (Valore 1961).

Very few studies have been conducted by researchers on the choice of type of mixer machine for use in preform foam concrete. Karl and Worner (1994) emphasized that the mixer should be capable of generating enough vertical motion of constituents in order to avoid the separation of light foam at top and heavier ingredients at bottom. When the conventional mortar mixer with blade rotating along central axis is used, the foam was observed to float above the mix due to improper mixing. Considering the above limitations, the pan mixer with rotating arm type is proved to be the best suitable mixing option in terms of quality due to its arm arrangement which can be located at various angles and facilitates both clock wise and anti-clock wise rotation (SathyaNarayanan and Ramamurthy

2013). In addition to pan mixer, paddle type mixer can also be recommended for preform method of production (ASTM C 796).

From the limited literature available on mix foam method of production it is found that a standard high speed paddle type mixer and pan mixer could be suitable options for mix foam concrete production (Akroyd 1962). It has been reported that a horizontal propeller type mixer having blade thickness of 0.75 inch and rotating speed of 55 to 60 rpm is generally preferred for cellular concrete preparation (The Aberdeen Group 1963). It is to be noted that speed and duration of mixing are very important parameters as they affect the stability of foam (Brady et al. 2001). Mixing of concrete for a very short duration of less than 2 minutes results in non-homogeneous mix (Valore 1961) while prolonged mixing greater than 6 minutes at high speed lead to disintegration of foam (Akroyd 1962). Hence, the dosage of foaming agent requirement to obtain a given density of concrete also depends on type of mixer used, length of mixing period and speed of mixing (Valore 1954a).

2.5 Fresh State Properties of Foam Concrete

In the fresh state, the foam concrete mix has a flowing and self compacting rheology. Hence the parameters such as consistency, stability and workability should be taken into account. Limited studies have been reported on these fresh concrete characteristics although several studies deal with properties of hardened foam concrete.

2.5.1 Consistency

Consistency refers to the ease with which concrete flows. Flow time using marsh cone and flow cone spread test are adopted to assess the consistency of foam concrete (Jones and McCarthy 2005a). Reportedly different factors related to mix compositions affects the consistency of the mix. Jones and McCarthy (2005a) compared the consistency and rheology of foam concrete made out of cement and unprocessed low lime coal fly ash with that of a cement-sand mix. It was reported that replacement of sand with unprocessed fly ash enhanced the consistency of mix which could be ascribed to the particle shape of fly ash particles. However use of fly ash affected the stability of foam and necessitated large foam volume to achieve the design density which was attributed to the high residual carbon in the ash. On the other hand, studies have reported that replacement of sand with fine fly ash can reduce the consistency of the mix due to presence of more amount of fines in the mix (Ramamurthy et al. 2009). Another important factor which affects consistency is the water content in the mix. Use of optimum water content is recommended, so as to achieve spreadability between 40% and 60% and flow time values below 20 sec, which have been identified as the reference workability for spreadable and flowable mixes that can be placed in the moulds without the aid of compaction or vibration (Nambiar and Ramamurthy 2008). It was also reported that consistency was reduced with increase in volume of foam due to reduced self weight and greater cohesion from higher air content (Karl and Worner 1994).

2.5.2 Stability

Different test methods have been proposed by researchers to measure the stability of the concrete mix. According to Nambiar and Ramamurthy (2008), the stability of foam concrete is the consistency at which the density ratio is nearly one (the measured fresh density/ design density) without any segregation and bleeding. The other approaches to assess the stability is to check the difference between calculated and actual quantities of foam required to achieve a plastic density within ± 50 kg/m^3 of the design value and to compare the calculated and actual water cement ratios. Inclusion of mineral admixtures such as ground granulated blast furnace slag (GGBS) and fly ash is also reported to affect the stability of foam and hence the stability of mix (Mellin 1999). Furthermore, incompatibility issues between super plasticizer (SP) and surfactant can also affect the stability of concrete mix. Hence from compatibility prospective, the dosage of SP in foam concrete was limited to 0.2% by weight of cement (Brady et al. 2001).

2.6 Physical Properties of Foam Concrete

Some physical properties of foam concrete such as density, air void characteristics and drying shrinkage have been discussed below.

2.6.1 Density

Many properties of lightweight concrete depend sensitively upon density of concrete. Density can be measured in two phases: fresh and dry densities. While specifying the density, the moisture conditions need to be indicated to represent the complete picture while comparing properties of foam concrete from different sources (Valore 1954b). Both the types of densities are useful at different stages. Fresh density is required for mix design and casting control while dry density significantly controls the mechanical, physical and durability related properties of hardened foam concrete. The density could be affected by many factors including type of fine aggregate, aggregate grading, type of foam, sand-cement ratio (McCormick 1967), foam agent volume (Bing et al. 2012) and supplementary cementitious materials (Nambiar and Ramamurthy 2006). McCormick (1967) reported that the density increases with increase in proportion of aggregates. Further it is reported that the fresh density decreases with an increase in foam volume content (Bing et al. 2012). The addition of fly ash as replacement for sand helps in reducing the density with an increased strength. The higher strength could be attributed to the reduction in foam volume requirement to achieve the design density due to lower specific gravity of fly ash (Nambiar and Ramamurthy 2006a).

2.6.2 Air void System

The pore system of foam concrete consists of water filled voids (gel and capillary pores) and artificially entrained or entrapped air voids of size greater than 50 micrometer. The pore structure is a very important characteristic as it influences the other material properties such as strength and durability. The pore structure is commonly characterized based on porosity, permeability and pore size distribution. Permeability which is the function of interconnectivity of pores eases the transport of fluid inside the hardened matrix of foam concrete. Hence the studies have proved that the water movement into concrete is not a simple function of porosity but depends on the pore diameter, distribution, continuity and tortuosity (Nambiar and Ramamurthy 2007a). Furthermore it is to be noted that the air void distribution is one of the most important properties influencing the strength of concrete. Foam concrete with narrower air void distribution shows higher strength.

Based on the studies, it is established that many factors can influence the microstructure of concrete such as mix composition, type of foaming agent and curing method. Adoption of very high water-cement ratio (w/c) results in increase in permeability and volume of larger pores (Hughes 1985; Mehta 1980). Also studies have proved that use of mineral admixtures such as fly ash and GGBS can result in pore refinement leading to further reduction in permeability (Gowripalan 1990). Adding to above, studies on use of fly ash as filler have established that uniform microstructure can be achieved in concrete. The other influential factor on the porosity is the volume of foaming agent. A majority of researchers reported that use of excessive foaming agent can result in bigger size bubble microstructure which can result in reduced strength accordingly. Furthermore, increase in curing temperature is found to result in significant reduction in porosity in mixes with fly ash as cement replacement material (Hassan et al. 1997).

2.6.3 Drying Shrinkage

One of the major deterrents to structural applications of foam concrete is the high drying shrinkage which has been stated to be up to 10 times greater than those observed in normal weight concrete. Reportedly different reasons contributing to such higher volume of shrinkage of foam concrete could be the use of higher amount of cement content and water content, less restraining effect of finer sand used and the absence of coarse aggregates in the mix composition (Jones et al. 2003; Valore 1954b). It is to be noted that the loss of water from comparatively bigger pores do not contribute much to shrinkage. In this line some experimental studies have revealed that the relatively lower shrinkage of mixes with higher foam volume is due to thinner pore wall and reduced volume of micro capillary pores which is distributed in this wall (Nambiar and Ramamurthy 2009; Nmai et al. 1997). However, Bing et al. (2012) presented a contradictory view stating that the increase in foam volume resulted in increase in shrinkage strain.

As an effect to reduce the shrinkage of foam concrete, use of light weight aggregate has been recommended as the aggregate plays an important role in restraining the shrinkage of cement matrix. On the other hand, use of alternative fine aggregates such as fly ash as sand replacement showed higher shrinkage than the corresponding cement sand mixes (Nambiar and Ramamurthy 2009). However, studies on use of fly ash as cement replacement material in foam concrete have proved that the use of 15 to 30% fly ash has resulted in significant reduction in shrinkage which could be attributed to the densification of matrix and reduction in porosity (Chindaprasirt and Rattanasak 2011; Giannakou and Jones 2002; Jones and McCarthy 2005b). Another crucial issue which affects the shrinkage the most is the high water demand of foam concrete. Hence the solution will be the suggestion of use of high range water reducing admixtures and shrinkage reducing admixtures to reduce the water demand and shrinkage of foam concrete. However, few studies have reported technical challenges in terms of non-compatibility issues such as foam collapse and slower rate of hydration which have limited the use of these chemical admixtures in foam concrete. Also use of fibres is reported to result in uniform microstructure and hence its inclusion is effective in reducing the drying shrinkage. In addition, the method of curing also plays a vital role with the autoclaving method leading to more crystalline tobermorite and thereby reduces the shrinkage significantly.

2.7 Mechanical and Durability Related Properties of Foam Concrete

2.7.1 Compressive Strength

Compressive strength is primarily a function of density of concrete and decreases exponentially with decrease in density. Other parameters which affect the strength of foam concrete are w/c ratio, sand particle type, curing method, cement sand ratio and type of foaming agent. The production of high strength foam concrete was reported when water/cement or binder ratio as low as 0.19 and 0.17 was adopted (Fujiwara et al. 1995). For a given density, the mix with fine sand exhibited higher strength than the mix with coarse sand. This is because, the addition of coarse sand, affect the pore size distribution in the paste (McCormick 1967; Nambiar and Ramamurthy 2006). Also cement replacements such as silica fume and fly ash affect the compressive strength of concrete significantly (Kearsley and Wainwright 2001). Studies have reported that up to 67% of the cement could be replaced with ungraded and graded fly ash without reducing the strength significantly. Also binary blend of silica fume and fly ash resulted in 25% enhancement of strength (Bing et al. 2012).

Inclusion of fibres also enhance the compressive strength of foam concrete by preventing the propagation of cracks and increasing the toughness of concrete. In terms of curing regime, autoclaving is generally adopted for pre cast elements and it increases the compressive strength. Kearsley and Visagie (1999) stated that a curing temperature of 40^0 C was optimum for enhancement of strength of foam concrete when fly ash is used as cement replacement material.

2.7.2 Water absorption and Sorptivity

The transport of aggressive liquid into concrete depends on its permeation characteristics like permeability, water absorption and sorption (Castro et al. 2011). Water absorption mainly representing the measure of total volume of pores in concrete. While the sorption is the measure of capillary pores of concrete to absorb the liquid by capillary action (Hall 1989). Hence sorptivity has been recognized as an important index of concrete durability because the test method reflect the way that most of the concrete is penetrated by water (Singh 2018). The sorption characteristics is observed to depend upon the pore structure, filler type and permeation mechanisms. Also a study by Jones and McCarthy (2005a) exploring the possibility of replacement of sand with fly ash has shown that mixes with fly ash resulted in marginally higher sorptivity than mixes with sand. Furthermore, it is reported that the permeation characteristics is mainly dependent on the volume of capillary pores in the paste phase and not all the entrained air voids are contributing to water permeation as most of them are not interconnected. Hence the water absorption and sorption is found to reduce with reduction in density of concrete (Nambiar and Ramamurthy 2007a).

2.7.3 Resistance to Aggressive Environment

Resistance to aggressive environment depend on the pore structure of foam concrete. Foam concrete with closed cellular structure can have good resistance to aggressive environment as the air voids can act as a buffer preventing rapid penetration. Sulphate resistance of foam concrete studied by (Jones 2001; Ranjani and Ramamurthy 2012) indicated that foam concrete has good resistance to aggressive chemical attack when compared to corresponding base mixes (mortar without foam). The resistance of foam concrete to carbonation was also assessed by few researchers. In a study by Jones and McCarthy (2005a) it was observed that lower density concrete carbonated at a relatively higher rate. On the contrary, it was discovered that the resistance to corrosion was found to be increasing with decrease in density in foam concrete samples (Chindaprasirt et al. 2008). From the review, it is evident that studies on long term durability behaviour of foam concrete is very limited.

2.8 FUNCTIONAL PROPERTIES OF FOAM CONCRETE

2.8.1 Thermal and Acoustic Insulation

From the limited literature available on the thermal performance of foam concrete, the thermal conductivity of foam concrete is found to depend on its microstructural parameters which includes the porosity, size and distribution of pore structure (Mydin 2011; Zhihua et al. 2005). It is well known that the air is the poor conductor of heat and hence, the more the air entrained, lesser the density of concrete, lower will be its thermal conductivity. Zhihua et al. (2005) reported a remarkable variation in thermal conductivity with the porosity of concrete. For instance, foam concrete of high dry density of 1490 kg/m^3 showed a thermal conductivity of 0.72 W/mK which dropped to as low as 0.16 W/mK for concrete with dry density as low as 430 kg/m^3. In a recent article, Zahari et al. (2009) have proved that the air voids which is of closed structure in foam concrete will suppress the heat flow and thus provide high thermal resistance. Furthermore, several researchers have reported that thermal conductivity is lesser for concrete with smaller pores than concrete with larger pores. This can be attributed to the prevention of heat transfer by convection by means of millions of tiny air pockets. Few researchers have also studied the effect of replacement of cement with fly ash on thermal behaviour of concrete. Giannakou and Jones (2002) reported 12 to 38% reduction in thermal conductivity of foam concrete with 30% pulverized fuel ash. The reduction in thermal conductivity is attributed to the cenospheric particle morphology and fly ash particles which resulted in increased heat flow path.

Recent studies show that foam concrete demonstrates higher sound insulation than normal concrete due to cellular microstructure (Narayanan and Ramamurthy 2000; Taylor 1967; VanDeijk 1919). While dense concrete tends to deflect the sound, foam concrete absorbs it. Hence foam concrete has 10 times higher rates of absorbing sound compared to dense concrete (Jones et al. 2003; Valore 1954b).

2.9 MAJOR GAPS IN RESEARCH WORK

Based on the review, it is observed that more systematic research is necessitated in the following areas as the available literature is limited. So far few researchers had reported that foam properties such as viscosity, foam density and foam stability can affect the cellular microstructure in concrete which in turn affect the mechanical and durability related properties of concrete (Amran et al. 2015, Brady et al. 2001, Kim and Jeong 2012). Furthermore, it is to be noted that foam stability is the most essential parameter for foam concrete production to ensure a uniform cellular structure of concrete as the bubble should remain stable till the concrete sets. Hence studies on foam stabilization mechanism to be carried out in particular to ensure uniform microstructure in hardened concrete. There are many studies which have extensively investigated the relationship between foam quality, bubble size and viscosity of surfactant solution (Bonsu et al. 2016). However, studies on correlation between foam properties and concrete properties are scarce. The nature of surfactant and charge carried by surfactants can also significantly affect the properties of foam. Recently, Falliano et al. (2018) based on a comparative study between synthetic and natural foam proved that the microstructure of foam concrete is very much dependent on nature of foaming agent. Hence more studies are needed in this direction. Also it is to be noted that the use of foam concrete has been increased over the past few years in most of the countries owing to its attributes such as low density, better thermal and sound insulation when compared to conventional concrete material. However, in India, its applications are very limited and foam concrete production hasn't gained confidence due to technical and engineering unfamiliarity and non-availability of economical foaming agents and foam generators. The above fact proves the need for identifying locally available and affordable foaming agents, which could make foam concrete production economical and facilitates its wider use. The difficulties encountered in the foam concrete production viz., mixing, transporting and pumping need to be addressed as they have major influence on the fresh and hardened properties of foam concrete. Another crucial issue is the high water demand requirement of foam concrete to maintain the self flowing nature and its stability. But the suggestion of use of water reducing admixture is also complicated as few studies have reported technical challenges in terms of non-compatibility issues such as foam collapse. Hence more investigations on issues on incompatibility between chemical admixtures (added to reduce the water demand and shrinkage of foam concrete) and foam need to be undertaken. Furthermore, there are no standard methods available to precisely determine the material mix proportion for foamed concrete which highlights the need for development of new mix design guideline for foam concrete. Also very limited literature is available on microstructure of foam concrete. More studies on improvement of microstructure of foam concrete is necessary as it has strong influence on material properties.

2.10 MOTIVATION FOR RESEARCH

It is evident that the use of foam concrete has been increased over the past few years in most of the countries owing to its attributes such as low density, better thermal and sound insulation when compared to conventional concrete material. However, in India, its applications are very limited and foam concrete production hasn't gained confidence due to technical and engineering unfamiliarity and non-availability of economical foaming agents and foam generators. Being a lightweight material with cellular structure, it is clearly evident that the pore structure of foamed concrete has an important influence on its properties and hence the selection of surfactant and the properties of foam are supposed to eventually affect the microstructure of concrete significantly. So far, few researchers had reported that foam properties such as viscosity, foam density and foam stability can affect the cellular microstructure in concrete which in turn affect the mechanical and durability related properties of concrete (Amran et al. 2015, Brady et al. 2001, Kim and Jeong 2012).

However, the investigations reported on the influence of behaviour of foam on the concrete properties are very limited.

For instance, Ranjani and Ramamurthy (2010) presented studies on the assessment of density and stability of foam produced with four different synthetic surfactants namely sodium lauryl sulfate, sodium lauryl ether sulfate, Sulfanol and cocodiethanolamide for use in foamed concrete production. Further studies conducted by Siva et al. (2015) established that addition of sodium admixtures such as NaCl, Na_2CO_3 and NaOH to surfactant Sodium lauryl sulphate resulted in improvement of foam properties which subsequently enhanced the performance in cement based mixes. Recently, Falliano et al. (2018) based on a comparative study between synthetic and natural foam proved that the microstructure of foam concrete is very much dependent on nature of foaming agent. Furthermore based on the studies on composite foaming agent, Ospanova et al. (2014) had reported that the addition of polyvinyl alcohol (PVA) as an additive to Sodium lauryl sulphate resulted in increased minimum equilibrium film thickness of film around bubble which eventually enhanced foam stability. From the above review on instances of different research, it is apparent that very limited work has been reported on influence of surfactant and foam behaviour on the properties of concrete. Therefore, there is an absolute necessity to explore new surfactants and additives for use in foam concrete production and to carry out systematic investigations on influence of surfactant and foam properties on foam concrete behaviour.

For the present study two different commercially available surfactants SLS (anionic), NPE (non-ionic) along with viscosity enhancing agent CMC have been used for foam production. One of the reasons for choice of surfactant SLS (anionic surfactant) and NPE (non-ionic surfactant) is to study the influence of charge of surfactants on the behaviour of foam and foam concrete. Although there are few studies reported earlier on foam concrete with SLS foaming agent which is relatively cheaper surfactant, however the initial foam density reported is as low as 26 kg/m^3. Hence there is a need for improvement of SLS foam. Previously it was reported by Colak (2000) based on his experimental studies that use of CMC as viscosity enhancing agent in foamed gypsum has resulted in increase in stability of the foam due to prevention of merging of bubbles. Hence it is decided in the present work to evaluate the suitability of CMC in performance improvement of foam produced with surfactant SLS. When NPE is considered, as of now, there is no research has been reported on use of NPE in foam concrete, though are few studies reported on use of NPE in foamed gypsum (Colak 2000). Therefore, the suitability of NPE and role of CMC in improvement of NPE foam for use in foam concrete production also need to be evaluated as a part of present study.

2.11 Objectives and Scope

The objectives of present study are:

i. To evaluate the relative performance of foam produced with identified synthetic surfactants and additives using different foam production methods through statistically designed experiments

ii. To ascertain the performance of synthetic surfactants and additive for use in foam concrete production by carrying out detailed investigations on relative workability, strength behavior, water absorption, sorptivity and air void characteristics of foam concrete. Also the correlation between properties of foam, surfactant and foam concrete properties will be investigated.

2.12 Scope

The scope of the study is limited to the following with respect to raw materials and methods adopted:

The study is restricted to moist cured foam concrete made of preform foaming method with the identified surfactants viz. sodium lauryl sulphate, nonyl phenol ethoxylated and additive carboxymethyl cellulose. As of now, there is no standard method for mix design of foam concrete available. However, ASTM C 796 has provided a method for calculation of foam volume required to make cement slurry of known water cement ratio and target density. Those equations are modified by including sand component to calculate foam volume for mortar. From the literature review it is observed that researchers have used c:s ratio of 1:1 (Ranjani 2011) and 1:2 (Nambiar 2006) for their studies. From economic point of view, a constant cement sand ratio 1:2 has been adopted for the present study. Further three different densities of foam concrete viz., 1000, 1500 and 1800 kg/m^3 were designed with foam volume ranging from 50 to 12% in order to study the influence of density on concrete properties. Ordinary Portland cement conforming to (IS 8112) and pulverized river sand finer than 300μm is used for the entire research work.

2.13 Summary of Literature Review

Based on the review conducted, it is observed that most of the investigations in the past have been confined to the evaluation of foam concrete properties alone rather on the characteristics of surfactant and foam itself which is more important to attain the desired properties of foam concrete. Further it is to be noted that the selection of surfactant, method of production of foam and foam production parameters play a vital role in the properties of foam. One of the important requirement of foam is its stability to ensure the uniform texture throughout the whole hardening process of concrete. Generally dry foam with lesser liquid fraction produced based on compressed air method is relatively stable and is recommended for foam concrete production. Foam stability is a function of various parameters such as thickness of lamellae, viscosity of liquid and bubble size. As per the guideline of ASTM C 796-97, the foam density should lie within the range of 32 to 64 kg/m^3 for preform foam concrete production. Among the characteristics of surfactant there is considerable interest in critical micelle concentration as it represents the lowest concentration at which surface active properties are at optimum. Foam generated with highly viscous surfactant solution is reported to have high initial foam density and stability over time which in turn resulted in desirable fresh and hardened state properties of foam concrete. From the studies on various classification of surfactant, it is observed that ionic surfactants perform better than non-ionic due to the electrical disjoining pressure at the interface. Presently, composite foaming agent perform better in terms of foamability and foam stability than the individual components due to synergistic effect.

In addition to surfactant and foam production parameters, mixing speed, duration of mixing and the type of mixer machine also greatly influenced foam concrete production. Based on the limited studies carried out on the influence of type of mixer machine, it is found that the pan mixer and paddle type mixer are the recommended options for foam concrete production. Also it is to be noted that there are no standard methods available to precisely determine the mix proportion design for foam concrete. Unlike the conventional (normal weight) concrete, wherein water-solids ratio is decided based on strength, in the case of foam concrete, the stability of mix governs the selection of water-solids ratio. Furthermore shrinkage being cement paste related phenomenon, the shrinkage strain of foam concrete which is basically a mortar is significantly greater than that of normal weight concrete. But this could be reduced by using light weight aggregates, high range water reducing admixtures and shrinkage reducing admixtures, fibres and by adopting autoclave method of curing. Foam concrete is reported to exhibit good thermal and sound insulation properties and these functional properties vary with its microstructural parameters. Also, studies have proved that foam concrete with closed cellular structure can have good resistance to aggressive environment as air voids act as buffer preventing rapid penetration.

Chapter 3
RESPONSE SURFACE MODELS ON BEHAVIOUR OF FOAM PRODUCED WITH DIFFERENT METHODOLOGIES

3.1 GENERAL

There have been several investigations in the past on the properties of foam concrete. Very little work has been reported in the literature on the influence of foam production parameters on foam characteristics for use in foam concrete production. It is reported in literature that the method of foam production has significant influence on the foam characteristics, which in turn can have great impact on concrete microstructure and other properties of concrete. Generally the manufacturer of foaming agent defines the foam production parameters such as dilution ratio and generation pressure for proprietary foaming agents and foam generator. However for new surfactants the above mentioned parameters need to determined through experimental investigations. This chapter is aimed at assessing the relative characteristics of foam produced with two different methodologies through systematic experiment design based on Response surface methodology.

3.2 MATERIALS AND METHODS

3.2.1 Materials Used

For the present study, the foam is produced by aerating commercially available synthetic surfactant namely sodium lauryl sulphate ($C_{12}H_{25}NaO_4S$). In order to improve the quality of foam, a viscosity enhancing agent namely carboxymethyl cellulose ($C_8H_{16}O_8$) is used as an additive along with base foaming agent.

3.2.2 Methods of Foam Production

In order to compare the influence of foam production methods on the behaviour of foam, two different foam production methods selected for the present study are mechanical method (stirrer) and compressed air method (foam generator). An electronic stirrer capable of varying frequency is used for thorough mixing of the surfactant solution (Fig. 3.1). As mentioned in earlier chapter, the speed of mixing is an important parameter for achieving good quality foam with stirrer.

The other equipment used for foam production is a laboratory based model of foam generator with the view of generating foam based on compressed air method. The entire set up comprises of two major components (i) air compressor (ii) foam generator. Fig. 3.2 shows the image of foam generator used for present study. Air compressor with high-pressurized air has an outlet valve connected to foam generator. The compressor is provided with a pressure regulator valve in order to control the pressure. In this method, foam is produced by forcing diluted surfactant solution through a series of high-density restrictions and at the same time forcing compressed air into the chamber present in control unit. The vigorous mixing action of chemicals along with water is carried out in a chamber of foam generator. The above mentioned thorough mixing is an important requirement for better performance of foam in terms of stability. The action of forcing pressurized air into the solution expands the foam and hence produce a thick or tight foam. The foam thus produced is conveyed out through a large outlet hose called as foam gun.

3.2.3 Parameters and Properties Studied

Surfactant (SLS) concentration, CMC concentration and foam generation pressure are the parameters considered. Surfactant (SLS) concentration and CMC concentration represents the concentration of chemical in the foaming premix solution prepared. For evaluating the relative characteristics of foam, a range of SLS concentration from 1% to 10% and CMC concentration from 0.1 % to 0.3% are adopted. Foam generation pressure refers to the pressure at which the foam is generated and it is varied from 353 kPa to 510 kPa. As mixing speed is important in case of stirrer method and hence for the present study, maximum speed of 1136 rpm is adopted for all trials as the preliminary studies established that foam performance is relatively good at higher speed.

The initial foam density refers to the density of foam measured immediately after its generation. As foam drainage is commonly used to characterize foam stability, for the present study, foam stability is assessed by free drainage test prescribed by Def-Standard 42-40. The foam drainage set up consists of a drainage pan of 1.612 litre nominal volume with a conical base. Polymethyl methacrylate tube of 12.7 mm internal diameter and 25 mm length with 1.6 mm diameter hole at its lower end is fitted to the conical base of drainage pan as shown in Fig. 3.3. This pan is filled with foam and the volume of the solution drained is measured at various time intervals.

Fig. 3.1 Hand stirrer (Method M1)

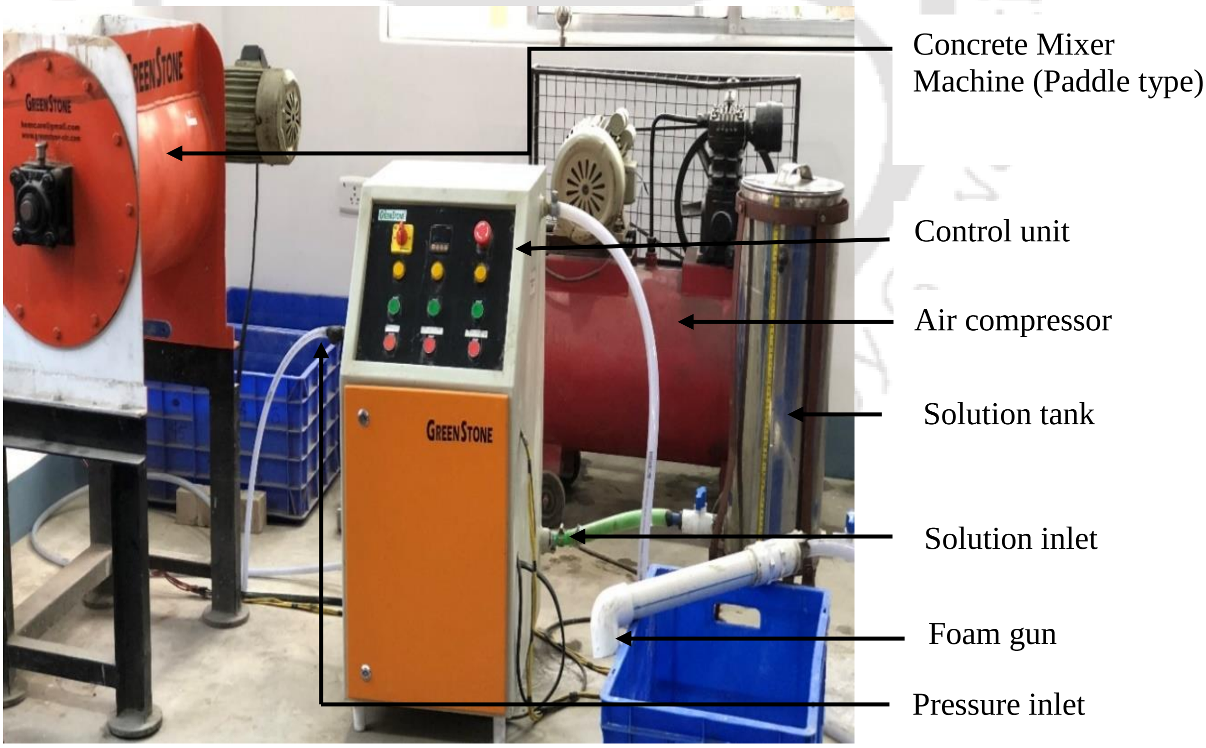
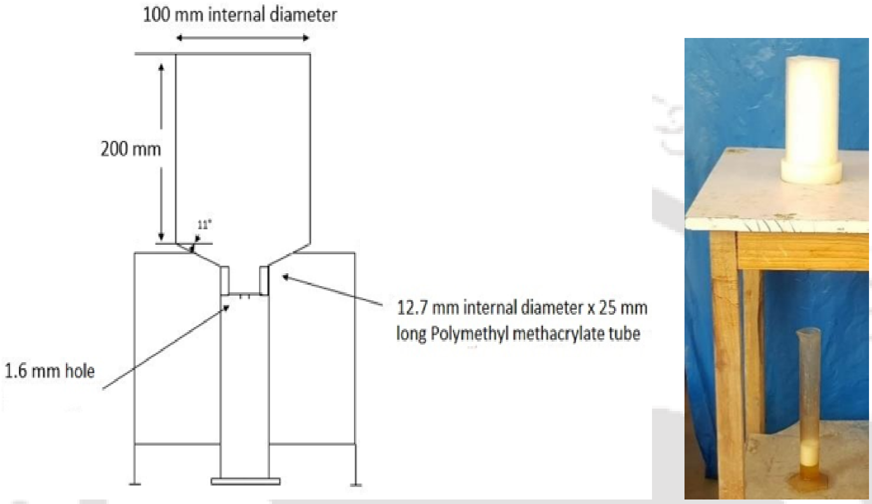


Fig. 3.2 Foam generator set up (Method M2)

The influence of addition of additive CMC on viscosity of surfactant solution is measured using Anton Par Rheometer with the speed of rotation varied from 150 to 200 rpm at constant temperature of 25^{0}C. Bubble microstructure is studied by placing a thin layer of foam on a glass plate (immediately after the generation of foam) under optical microscope and the images captured are analyzed with a computer with image analysis software (Image J 2006).



(a) (b)

Fig. 3.3 Foam drainage set-up

3.2.4 Preliminary Studies to Optimize the Speed of Stirrer

Fig. 3.4 shows the variation of initial foam density of foam with the speed of stirrer. As mixing speed is important in case of stirrer method, studies on influence of speed of stirrer on initial foam density has been carried out for different surfactant and CMC concentrations as presented in Fig. 3.4. Based on the experimental outcomes it is observed that irrespective of surfactant and CMC concentration, the initial foam density of foam always reduces with increase in speed of stirrer. Therefore, in order to produce foam with relatively lesser liquid fraction with stirrer, it is recommendable to use higher speed. Hence for the present study, maximum speed of 1136 rpm is adopted for all the trials with stirrer.

3.2.5 Experimental Design

Based upon the preliminary studies carried out on properties of foam using single factor experiments the range of parameters are fixed up for further optimization studies based on Response Surface Methodology (RSM). Basically RSM is a statistical technique intended for process optimization where it can be used for various aspects starting from modelling and analysis of problems with various factors associated with it (Montgomery 2001).

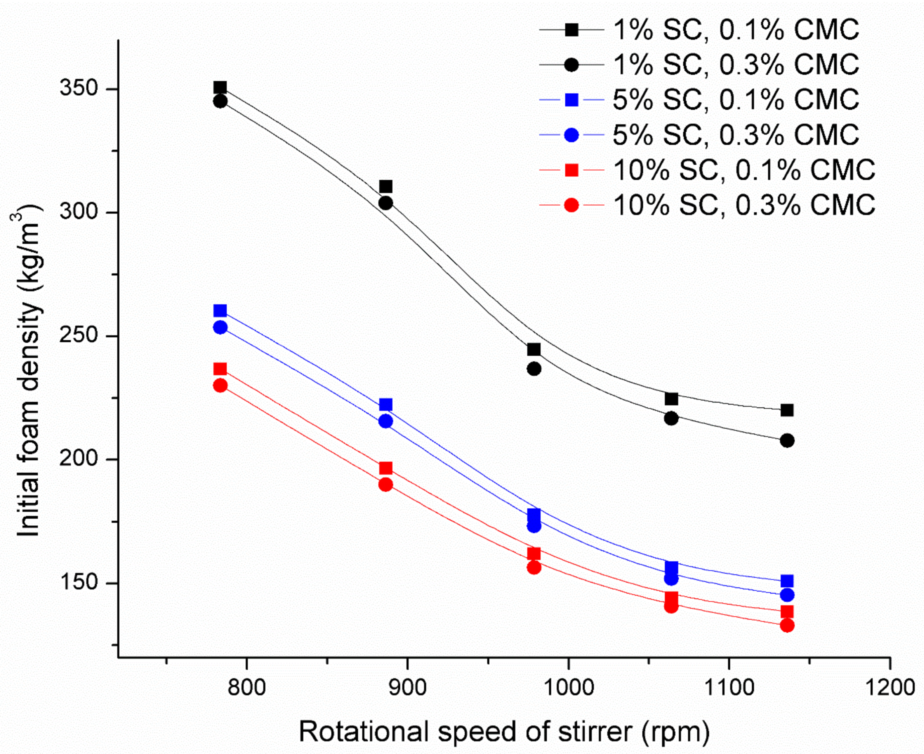


Fig. 3.4 Variation of initial foam density with rotational speed of stirrer

Hence for the present study, a special case of factorial design based on response surface methodology (RSM) using central composite design (CCD) is adopted due to its special features of rotatability and equal precision of estimates in all the directions. A design matrix is formulated to study the effect of two independent variables for stirrer model (M1) namely x_1 SLS concentration (SLS), x_2 CMC concentration on four response variables namely initial foam density (IFD), foam stability (FS), viscosity of surfactant solution and average bubble diameter respectively (Table 3.1 and Table 3.2). In a similar fashion, a design matrix is formulated for generator model (M2) with three independent variables x_1, x_2 and x_3 (foam generation pressure) to study the effect on four response variables as mentioned above (Table 3.3 and Table 3.4). For each surfactant thirteen experimental treatments for M1 (Table 3.2) and twenty experimental treatments for M2 (Table 3.4) are assigned based on the CCD with two levels of each variable for M1 and M2 respectively. A CCD has three groups of design points, which includes factorial point, axial point and centre point. The factorial points contribute to the estimation of the interaction terms. The centre points provide information about the existence of curvature in the system, with which the axial points allow to estimate the quadratic terms (Sotoperez et al. 2015). The centre point "C" is repeated several times according to the experimental design and the experiments were randomized in order to minimize the effects of unexplained variability in the actual responses due to extraneous factors. CCD can have different design properties by controlling the value of α which is the distance from each axial point to the centre of the design space. The value of α can be calculated using the formulae given below.

$$\alpha = (2^k)^{0.25} \tag{3.1}$$

Where k is the number of points used in the factorial portion of the design. The experimental design matrix, data analysis and optimization procedure are performed using Design Expert Software (version 10.0.6). Multiple regression coefficients are determined by employing least square technique to predict the polynomial models for the response variables studied. The generalized polynomial model proposed for predicting the response variables for M2 is given below:

$$Yi = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} {x_1}^2 + \beta_{22} {x_2}^2 + \beta_{33} {x_3}^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \tag{3.2}$$

Where Yi is the predicted response, β_0 is constant, β_1 , β_2 and β_3 are regression coefficients for main variables, β_{11} , β_{22} and β_{33} are quadratic effects and β_{12} , β_{13} and β_{23} are interaction effect or cross product coefficient of independent variables. The main effect refers to the change in response produced by a change in level of the factor while the interaction effects represents the combined effects of variables on the criteria. When an interaction effect is present, the impact of one variable depends on the level of the other variable. Generally, when an interaction is large the corresponding main effects have little practical meaning.

Table 3.1 Factors and factor levels adopted for design matrix (Model M1)

Notation	Factor	Low level	High level
SC	SLS Concentration (%)	1	10
CMC	CMC Concentration (%)	0.1	0.3

Table 3.2 Matrix of the central composite design (Model M1)

Treatment runs	SLS concentration (%)	CMC concentration (%)
1	1	0.2
2	10	0.2
3	5.5 (C)	0.2
4	1	0.3
5	5.5 (C)	0.2
6	10	0.3
7	5.5 (C)	0.2
8	1	0.1
9	10	0.1
10	5.5 (C)	0.2
11	5.5	0.3
12	5.5 (C)	0.2
13	5.5	0.1

C : Centre point

Table 3.3 Factors and factor levels adopted for design matrix (Model M2)

Notation	Factor	Low level	High level
SC	SLS Concentration (%)	0.5	10
CMC	CMC Concentration (%)	0.1	0.3
FGP	Foam generation pressure (kPa)	353	510

3.3 Regression Analysis

The results are analyzed using the Design Expert Software (version 10.0.6) to determine the quadratic response surface models. The quadratic response surface models (RSM) are used to assess the relative performance of foam produced with surfactant SLS and additive CMC. The details of model equations for initial foam density, foam stability (from drainage study at various time intervals after foam generation), viscosity of foaming solution and average bubble diameter are presented in Table 3.5 and Table 3.6. Though the viscosity of surfactant solution is independent of method of foam generation, the model equation for viscosity is presented under method M1 just for the convenience in presentation.

Table 3.4 Matrix of the central composite design (Model M2)

Treatment runs	SLS concentration (%)	CMC concentration (%)	Foam generation pressure (kPa)
1	5.25	0.3	431
2	2.42	0.25	384.42
3	5.25	0.2	353
4	5.25 (C)	0.2	431
5	0.5	0.2	431
6	5.25 (C)	0.2	431
7	5.25	0.2	510
8	5.25	0.1	431
9	10	0.2	431
10	8.07	0.25	384.42
11	2.42	0.25	477.58
12	2.42	0.14	384.42
13	8.07	0.25	477.58
14	8.07	0.14	384.42
15	5.25 (C)	0.2	431
16	5.25 (C)	0.2	431
17	2.42	0.14	477.58
18	5.25 (C)	0.2	431
19	8.07	0.14	477.58
20	5.25 (C)	0.2	431

C : Centre point

3.3.1 Effect of Factors and Their Interaction

The significance of the estimated regression coefficients for each response variable is assessed by F- ratio at a probability of 0.05. As shown in Table 3.7 and Table 3.8, the main effects of independent variables appear to have the most significant effect ($p < 0.05$) as compared to quadratic and interaction effects. It is also observed that all main effects exhibits positive effect on response variable which indicates that an increase in factor level is associated with an increase in response variable.

Table 3.5 RSM for different properties of foam and surfactant (Stirrer method M1)

Properties	Response Surface Models
Initial foam density (kg/m^3)	+227.90 - (21.276*SC) - (28.497*CMC) - (1.049*SC*CMC) +(1.27*SC^2) + (27.972*CMC^2)
Foam drainage at 5th min (%)	+56.978 - (4.343* SC) - (60.80*CMC) + (1.428*SC*CMC) +(0.137*SC^2) + (37.278*CMC^2)
Foam drainage at 10th min (%)	67.97 - (4.214* SC) - (61.83*CMC) + (2.62*SC*CMC) +(0.169*SC^2) + (51.176*CMC^2)
Foam drainage at 15th min (%)	75.901 - (3.175* SC) - (94.325*CMC) + (0.212*SC*CMC) +(0.1*SC^2) + (151.75*CMC^2)
Viscosity (mPa-sec)	+3.706 - (0.143*SC) - (15.207*CMC) + (1.50*SC*CMC) + (7.56*10^{-003}*SC^2) + (106.31*CMC^2)
Average bubble diameter (µm)	+204.813 - (6.876*SC) - (320.814*CMC) + (10.555*SC*CMC) +(0.126*SC^2) + (406.896*CMC^2)

3.3.2 Model Adequacy Check

The adequacy of response models are determined using model analysis, coefficient of determination (R^2) analysis and by comparing the experimental data with values predicted by response surface models (Montgomery 2001). Validation of the second order polynomial regression models with additional experimental data are observed to be highly adequate to interpret a reliable relationship between the independent and response variables with a satisfactory coefficient R^2 (> 0.95) for most of the regression models (Table 3.9 and Table 3.10). Adequacy of the model is also checked by examining the residuals for trends by looking at the diagnostic plots like normal probability plots and residual plots (Fig. 3.5 to Fig. 3.10)

Table 3.6 RSM for different properties of foam (Generator method M2)

Foam properties	Response Surface Models
Initial foam density (kg/m^3)	+165.673 - (5.062*SC) + (56.33*CMC) - (24.86*FGP) + (0.766*SC*CMC) + (0.22*SC*FGP) - (14.186*CMC*FGP) + (0.217*SC^2) - (124.586*CMC^2) + (0.53*FGP^2)
Foam drainage at 5th minute (%)	+9.535 + (0.962*SC) - (15.90*CMC) + (1.55*FGP) - (1.77*SC*CMC) - (0.192*SC*FGP) - (0.79*CMC*FGP) - (0.027*SC^2) + (30.90*CMC^2) - (0.288*FGP^2)
Foam drainage at 10th minute (%)	+118.78 + (0.65*SC) - (417.921*CMC) + (3.943*FGP) - (2.419*SC*CMC) - (0.24*SC*FGP) +(73.08*CMC*FGP) - (0.184*SC^2) + (40.56*CMC^2) - (3.511*FGP^2)
Foam drainage at 15th minute (%)	+192.57 + (5.92*SC) - (542.908*CMC) - (16.694*FGP) - (4.71*SC*CMC) - (1.54*SC*FGP) +(75.11*CMC*FGP) - (0.218*SC^2) + (273.81*CMC^2) - (0.795*FGP^2)
Average bubble diameter (μm)	+636.218 - (2.95*SC) - (1372.209*CMC) - (104.915*FGP) - (1.488*SC*CMC) - (1.674*SC*FGP) +(114.904*CMC*FGP) + (0.324*SC^2) + (1532.876*CMC^2) + (6.763*FGP^2)

Table 3.7 ANOVA and regression coefficients of response surface models fitted (Stirrer method M1)

Variables		Main effects		Quadratic effects		Interaction effect
		x_1	x_2	${x_1}^2$	${x_2}^2$	$x_1\,x_2$
Initial foam density (kg/m^3)	p- value	<0.0001	<0.0001	<0.0001	0.447	0.145
	F value	20601.03	96.01	5487.77	0.65	2.68
Foam drainage at 5th minute (%)	p- value	<0.0001	0.0014	0.0382	0.7434	0.5024
	F value	237.56	26.24	6.49	0.12	0.50
Foam drainage at 10th minute (%)	p- value	<0.0001	0.0009	0.0021	0.5028	0.0910
	F value	279.06	30.05	22.46	0.50	3.84
Foam drainage at 15th minute (%)	p- value	<0.0001	0.0048	0.1279	0.2376	0.9247
	F value	131.29	16.56	2.98	1.67	$9.61*10^{-003}$
Viscosity (mPa-sec)	p- value	<0.0001	<0.0001	0.4264	0.0006	0.0029
	F value	76.59	835.75	0.71	34.37	20.07
Average bubble diameter (µm)	p- value	<0.0001	<0.0001	0.2074	0.0637	0.0175
	F value	146.12	63.52	1.93	4.84	9.55

As it can be seen from Fig. 3.5 to Fig. 3.10 the normality assumption is satisfied as the residuals plot approximately along the straight line. Also the plots of residuals versus the predicted responses shows that all the data points were randomly distributed showing that the variance is constant. Furthermore, a few additional tests are conducted to confirm the accuracy of prediction relations, where the difference between predicted and observed responses are found to be within 10% for most of the trials as shown in Table 3.11 and Table 3.12.

Table 3.8 ANOVA and regression coefficients of response surface models fitted (Generator method M2)

Variables		Main effects			Quadratic effects			Interaction effect		
		x_1	x_2	x_3	x_1^2	x_2^2	x_3^2	$x_1\ x_2$	$x_1\ x_3$	$x_2\ x_3$
Initial foam density(kg/m^3)	p-value	0.0001	0.0026	< 0.0001	0.0441	0.5720	0.8750	0.9013	0.7757	0.7000
	F value	36.41	15.88	179.44	5.30	0.34	0.026	0.016	0.086	0.16
Foam drainage at 5th minute (%)	p-value	<0.0001	<0.0001	<0.0001	0.0626	0.3153	0.5421	0.0573	0.0918	0.8748
	F value	194.88	83.75	92.84	4.39	1.12	0.40	4.61	3.48	0.026
Foam drainage at 10th minute (%)	p-value	<0.0001	<0.0001	<0.0001	0.1021	0.8641	0.3535	0.7186	0.7706	0.0886
	F value	91.29	43.31	59.69	3.24	0.031	0.95	0.14	0.090	3.56
Foam drainage at 15th minute (%)	p-value	<0.0001	0.0020	0.0014	0.3520	0.5997	0.9218	0.7482	0.4070	0.3963
	F value	40.04	17.10	18.92	0.95	0.29	0.010	0.11	0.75	0.79
Viscosity (mPa-sec)	p-value	<0.0001	<0.0001	-	0.4264	0.0006	-	0.0029	-	-
	F value	76.59	835.75	-	0.71	34.37	-	20.07	-	-
Average bubble diameter (μm)	p-value	<0.0001	0.0006	0.0008	0.4192	0.1085	0.6295	0.9529	0.5977	0.4494
	F value	41.51	24.18	22.07	0.71	3.10	0.25	$3.668*10^{-003}$	0.30	0.62

Table 3.9 R^2, Probability value and F values for the final models (Stirrer method, M1)

Response variable	R^2	R^2 adj	Regression p value	F value
Initial foam density (kg/m^3)	0.9997	0.9996	< 0.0001	5434.61
Foam drainage at 5th minute (%)	0.9750	0.9571	< 0.0001	54.56
Foam drainage at 10th minute (%)	0.9800	0.9657	< 0.0001	68.56
Foam drainage at 15th minute (%)	0.9569	0.9261	0.0001	31.06
Viscosity (mPa-sec)	0.9929	0.9878	< 0.0001	195.57
Average bubble diameter (μm)	0.9704	0.9493	<0.0001	45.97

Table 3.10 R^2, Probability value and F values for the final models (Generator method, M2)

Response variable	R^2	R^2 adj	Regression p value	F value
Initial foam density (kg/m^3)	0.9597	0.9234	< 0.0001	26.44
Foam drainage at 5th minute (%)	0.9747	0.9520	< 0.0001	42.88
Foam drainage at 10th minute (%)	0.9529	0.9104	< 0.0001	22.46
Foam drainage at 15th minute (%)	0.8877	0.7867	0.0011	8.78
Viscosity (mPa-sec)	0.9929	0.9878	< 0.0001	195.57
Average bubble diameter (μm)	0.9023	0.8143	0.0006	10.26

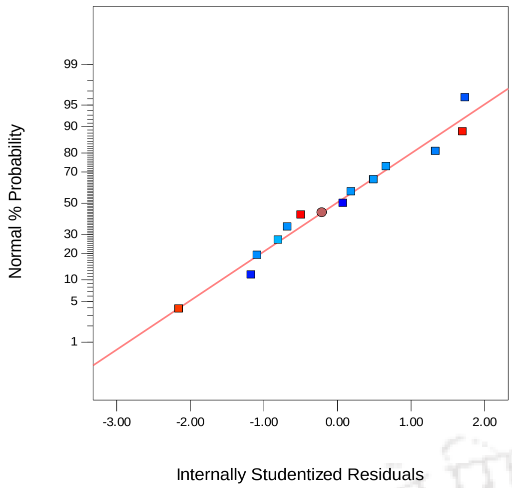


Fig 3.5 (a) Normal probability plot (M1)

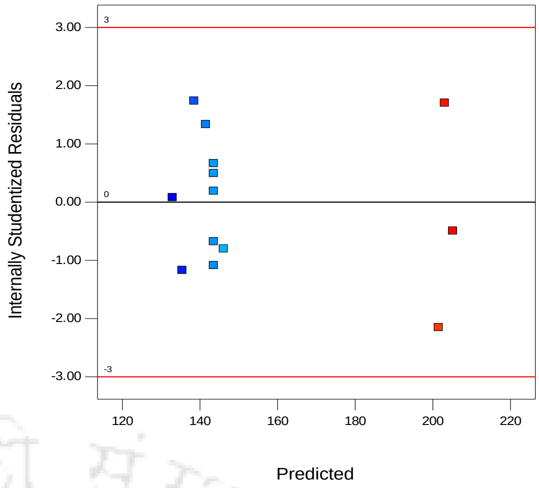


Fig 3.5 (b) Residual plot (M1)

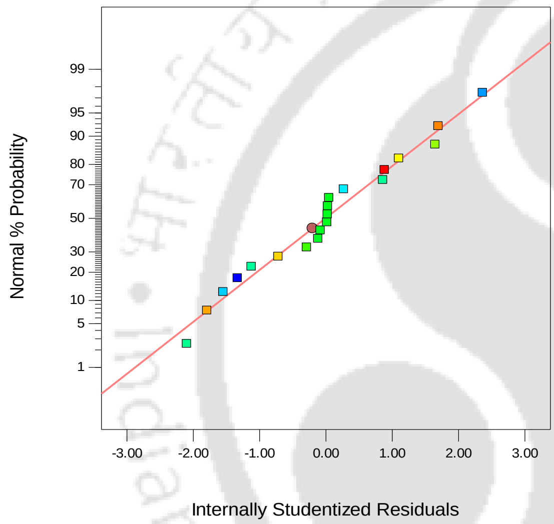


Fig 3.5 (c) Normal probability plot (M2)

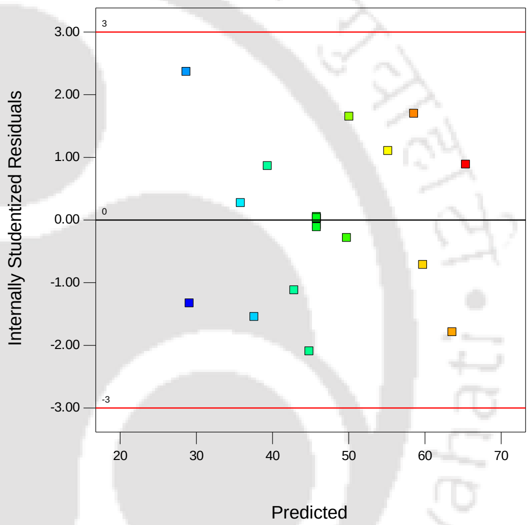


Fig 3.5 (d) Residual plot (M2)

Fig. 3.5 Diagnostic plots for initial foam density

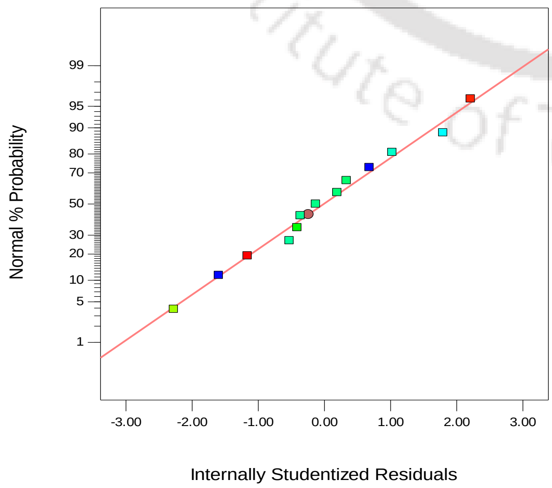


Fig 3.6 (a) Normal probability plot (M1)

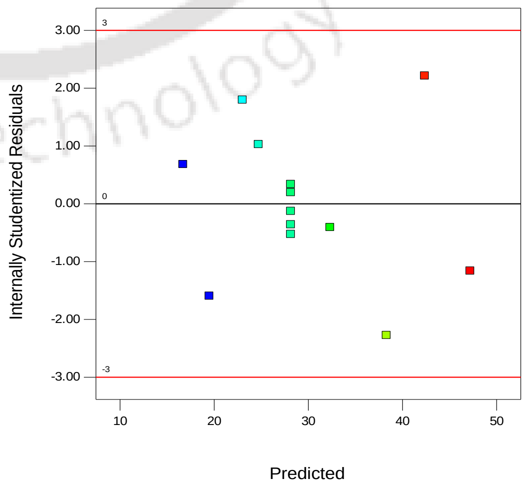


Fig 3.6 (b) Residual plot (M1)

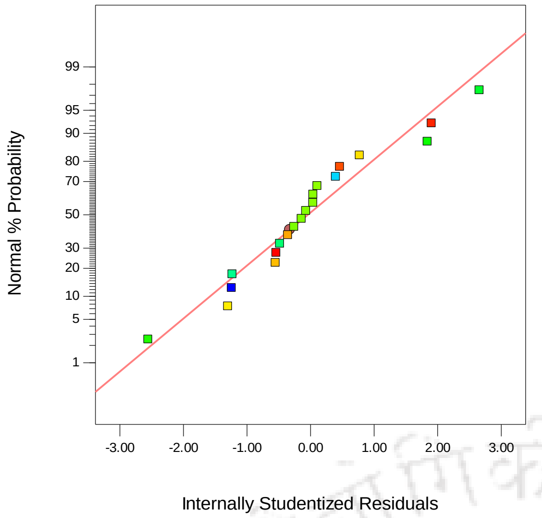


Fig 3.6 (c) Normal probability plot (M2)

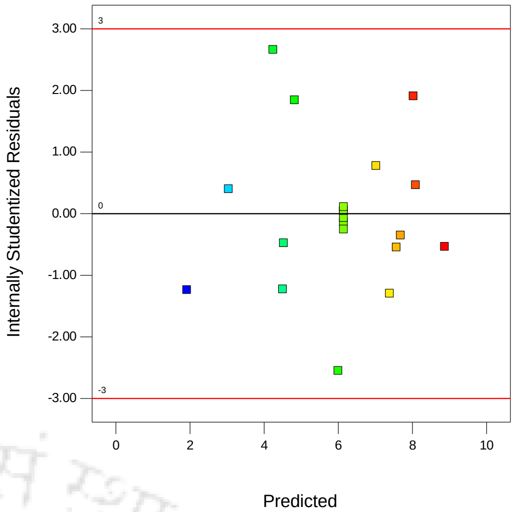


Fig 3.6 (d) Residual plot (M2)

Fig. 3.6 Diagnostic plots for % solution drained at 5th minute after foam generation

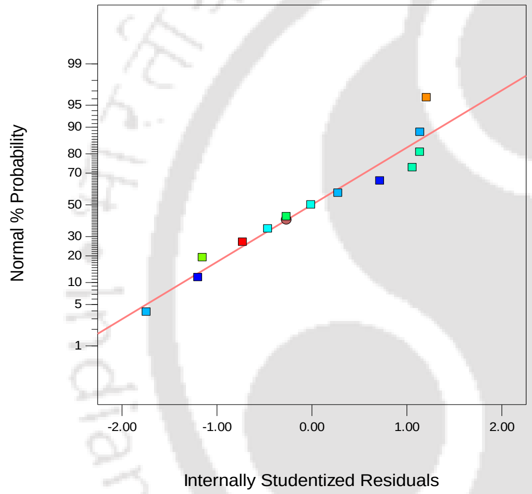


Fig 3.7 (a) Normal probability plot (M1)

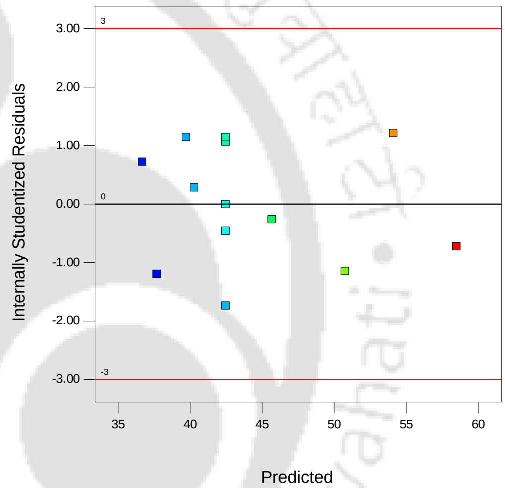


Fig 3.7 (b) Residual plot (M1)

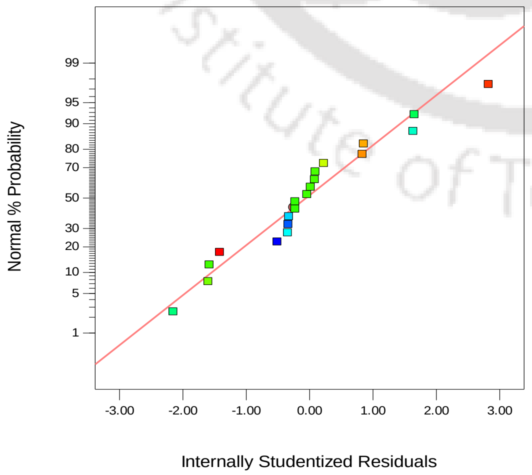


Fig 3.7 (c) Normal probability plot (M2)

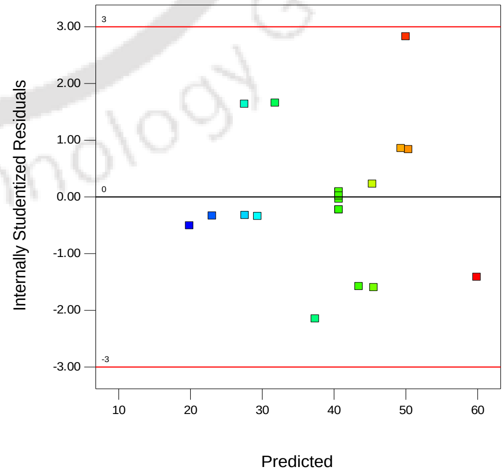


Fig 3.7 (d) Residual plot (M2)

Fig. 3.7 Diagnostic plots for % solution drained at 10th minute after foam generation

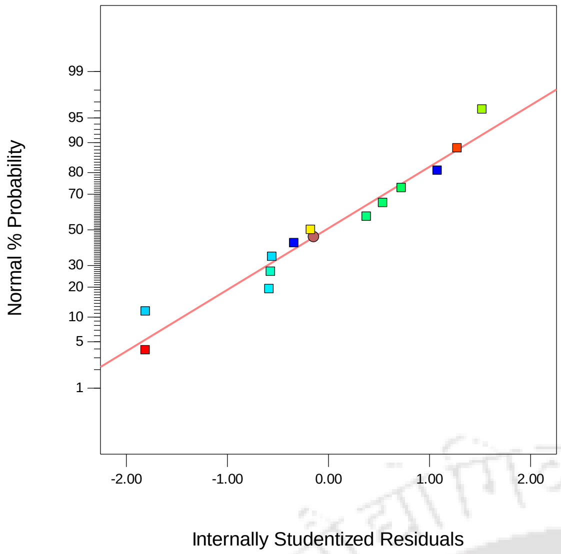


Fig 3.8 (a) Normal probability plot (M1)

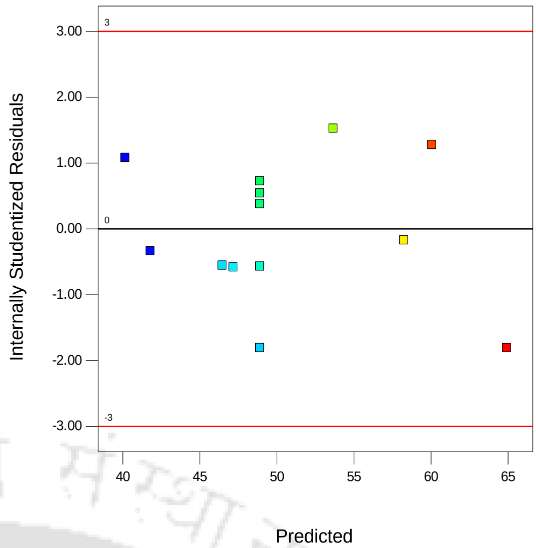


Fig 3.8 (b) Residual plot (M1)

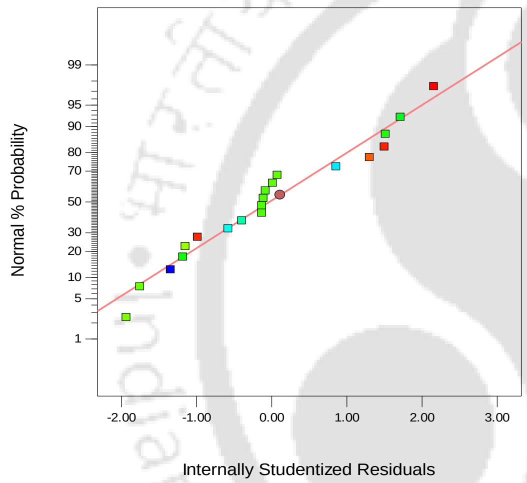


Fig 3.8 (c) Normal probability plot (M2)

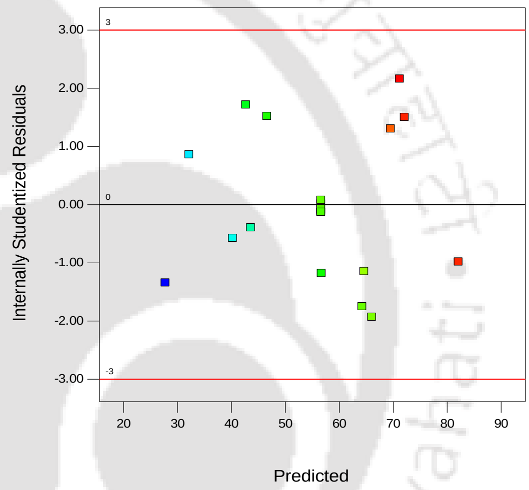


Fig 3.8 (d) Residual plot (M2)

Fig. 3.8 Diagnostic plots for % solution drained at 15th minute after foam generation

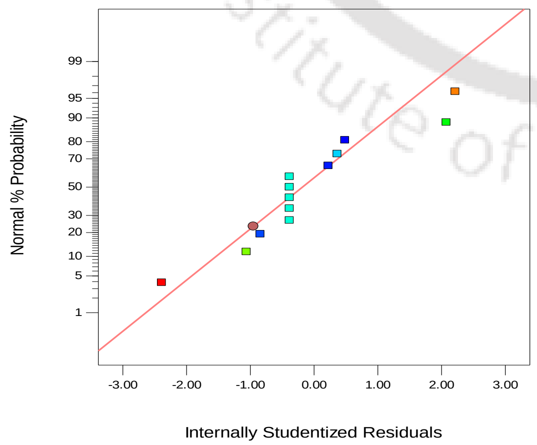


Fig 3.9 (a) Normal probability plot

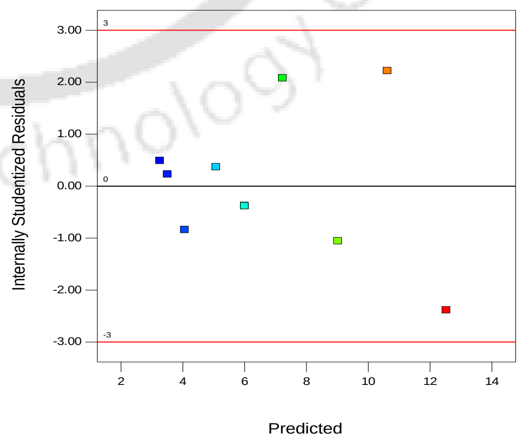


Fig 3.9 (b) Residual plot

Fig. 3.9 Diagnostic plots for viscosity of surfactant solution

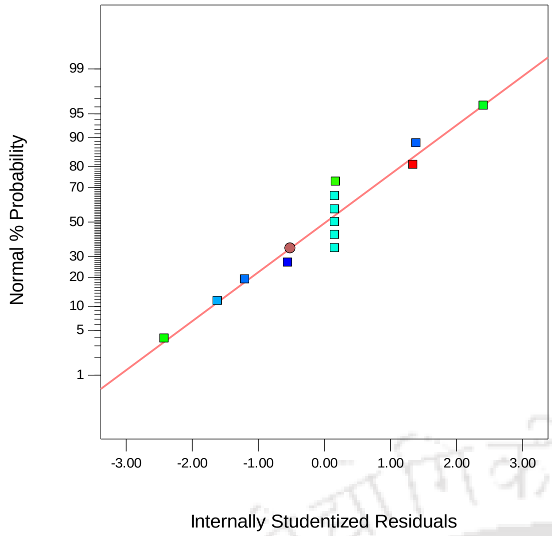


Fig 3.10 (a) Normal probability plot (M1)

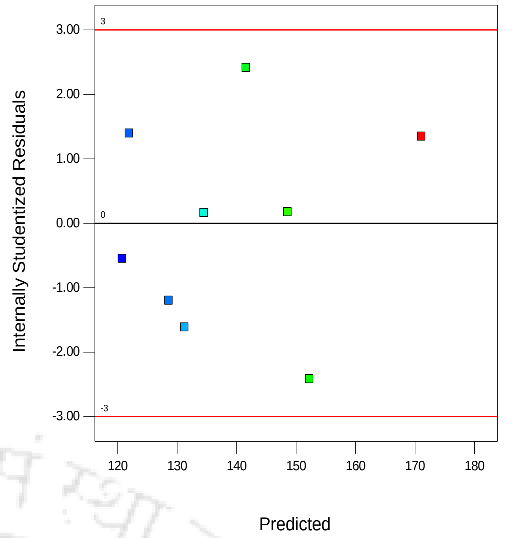


Fig 3.10 (b) Residual plot (M1)

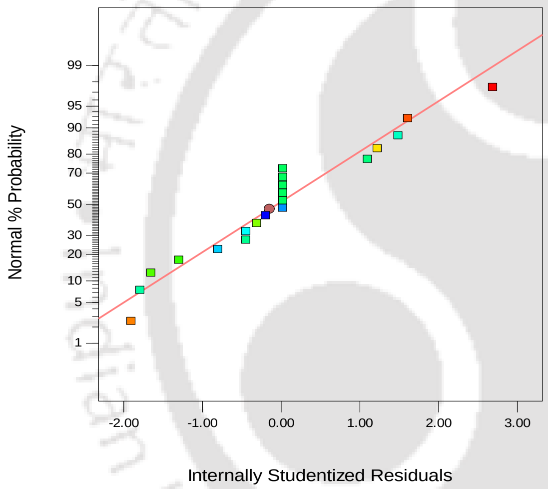


Fig 3.10 (c) Normal probability plot (M2)

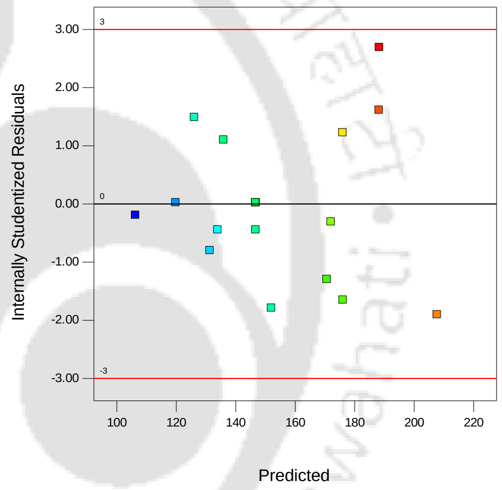


Fig 3.10 (d) Residual plot (M2)

Fig. 3.10 Diagnostic plots for average bubble diameter of foam

Table 3.11 Observed and predicted responses for confirmation of models (SLS, stirrer method M1)

Sl	Factors		Initial foam density (kg/m^3)			Solution drained at 5^{th} minute (%)			Solution drained at 10^{th} minute (%)			Solution drained at 15^{th} minute (%)			Viscosity (mPa-sec)			Average bubble diameter (μm)		
	SLS (%)	CMC (%)	P	O	D	P	O	D	P	O	D	P	O	D	P	O	D	P	O	D
1	4	0.2	157.70	150.82	6.88	32.28	31.4	0.88	45.6	45.83	-0.23	52.17	53.2	-1.03	5.66	5.21	0.45	139.89	147	-7.11
2	1	0.1	205.22	204.09	1.13	47.2	50.27	-3.07	58.51	60.95	-2.44	64.93	69.78	-4.85	3.26	3.12	0.14	171.1	168.78	2.32
3	7	0.1	137.90	134	3.90	28.61	28	0.61	42.94	44.83	-1.89	50.81	49.3	1.51	3.66	4.02	-0.36	142.26	150.6	-8.34
4	2.5	0.1	179.82	168.30	11.52	41.63	38.25	3.38	53.47	49.7	3.77	60.72	56.91	3.81	3.31	3.14	0.17	163.04	157.64	5.40
5	4	0.1	160.13	154.21	5.92	36.67	35.2	1.47	49.2	46.18	3.02	56.97	55.24	1.73	3.39	3.11	0.28	155.54	159	-3.46
6	7	0.2	135.16	138	-2.84	24.65	27.1	-2.45	40.13	43.47	-3.34	46.08	48	-1.92	6.38	6.86	-0.48	129.78	135	-5.22
7	2.5	0.3	175.83	169	6.83	33.16	36.13	-2.97	46.51	47.35	-0.84	54.1	55.5	-1.40	9.52	9.92	-0.40	136.7	129	7.70

P – Predicted value

O – Observed value

D – % Difference = [(Predicted – Observed)/ Predicted]*100

Table 3.12 Observed and predicted responses for confirmation of models (SLS, Generator method M2)

Sl	Factors			Initial foam density (kg/m^3)			Solution drained at 5th minute (%)			Solution drained at 10th minute (%)			Solution drained at 15th minute (%)			Average bubble diameter (μm)		
	SC (%)	CMC (%)	FGP (kPa)	P	O	D	P	O	D	P	O	D	P	O	D	P	O	D
1	0.5	0.2	431	58.58	61.62	-5.19	8.03	8.5	-5.85	50.01	55	-9.98	71.24	77.41	-8.66	210.31	208	1.10
2	3	0.14	384.4	62.07	60.39	2.71	8.66	8.8	-1.62	57.95	59.5	-2.67	79.78	77.28	3.13	200.4	202	-0.80
3	2.5	0.2	412	56.5	58.58	-3.68	7.7	6.99	9.22	49.54	48.72	1.66	68.76	67.45	1.91	174.6	160	8.36
4	4	0.3	510	23.22	25.07	-7.97	3.88	3.52	9.28	28.31	28.66	-1.24	45.63	50.12	-9.84	135.86	149	-9.67
5	9.5	0.1	471	38.63	39.24	-1.58	4.92	5.25	-6.71	26.66	27.29	-2.36	40.25	37.51	6.81	145.21	158	-8.81
6	5.25	0.2	373	59.08	58.95	0.22	7.33	7.42	-1.23	47.59	47.55	0.08	66.42	71.49	-7.63	167.89	178	-6.02
7	8.074	0.14	384.4	53.62	57	-6.30	6.85	6.23	9.05	44.24	39.96	9.67	62.86	66.16	-5.25	168.63	155	8.08

P – Predicted value

O – Observed value

D – % Difference = [(Predicted – Observed)/ Predicted]*100

3.3.3 Response Surface Model for Initial Foam Density

The response surface plots for initial foam density of foam produced with stirrer (Method M1) and foam generator (Method M2) are shown in Fig. 3.11. The initial foam density of foam produced using stirrer (Method M1) decreases significantly with increase in SLS concentration up to 7%, beyond which there is not much variation. For instance, when SLS concentration is varied from 1 to 10 % for a constant CMC concentration of 0.3%, a significant reduction of 34% in initial foam density is observed. The reason for this behavior can be ascribed to the production of foam with relatively good quality (more amount of air and lesser liquid fraction) at higher SLS concentration. However, the increase in CMC concentration from 0.1 to 0.3% for a constant SLS concentration of 1% do not result to significant variation in initial foam density. Hence the variation in initial foam density with CMC concentration is very less compared to that with SLS concentration.

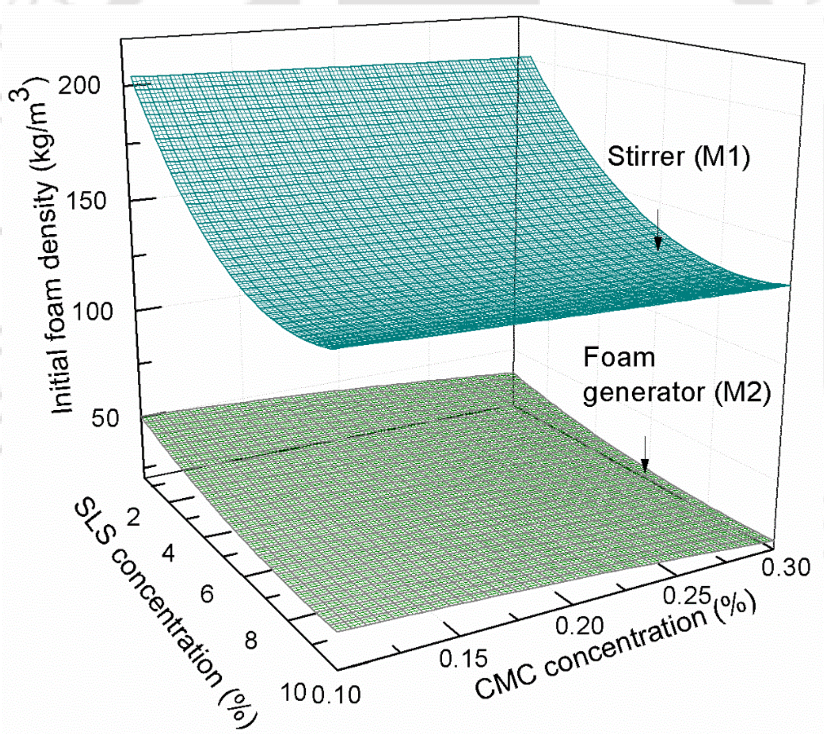


Fig. 3.11 Response surface plots for initial foam density for M1 and M2 (FGP 490 kPa)

The variation of initial foam density of foam with SLS concentration produced using foam generator (Method M2) also shows decreasing trend with increase in SLS concentration similar to that of foam produced using method M1. However, it is to be noted that the variation observed is linear for method M2 while it is non-linear for method M1 (Fig. 3.11). For instance, a reduction of 45% in initial foam density is observed when CMC concentration is increased from 0 to 0.3% at 1% SLS concentration for generator method M2 (Fig. 3.12). This shows that the potential of CMC in improvement of quality of foam (reduction of liquid fraction) is utilized to a greater extent in foam produced using foam generator based on compressed air method.

As the foam produced using method 2 has 3 factors, the effect of FGP on initial foam density is studied from the response surface plot as shown in Fig. 3.13. For a given SLS concentration, an increase in foam generation pressure reduces the initial foam density significantly for foam produced using method M2 (Fig. 3.13). Also it is to be noted that the effect of foam generation pressure is very predominant when compared to other parameters for foam produced using method M2. Hence for the foam generator model used for the present study, to produce foam of good quality (lower liquid fraction) higher pressure is needed for sufficient mixing of air and liquid. Further, the experimental results indicates that the foam produced using generator is relatively dry foam and meets ASTM C 796 requirement of initial foam density of 32 to 64 kg/m^3 at the optimum levels of foam production parameters as discussed in chapter 4. Based on the above observations, it can be concluded that to produce good quality foam (lower liquid fraction), it is preferable to use higher levels of SLS and CMC concentration in addition to higher foam generation pressure.

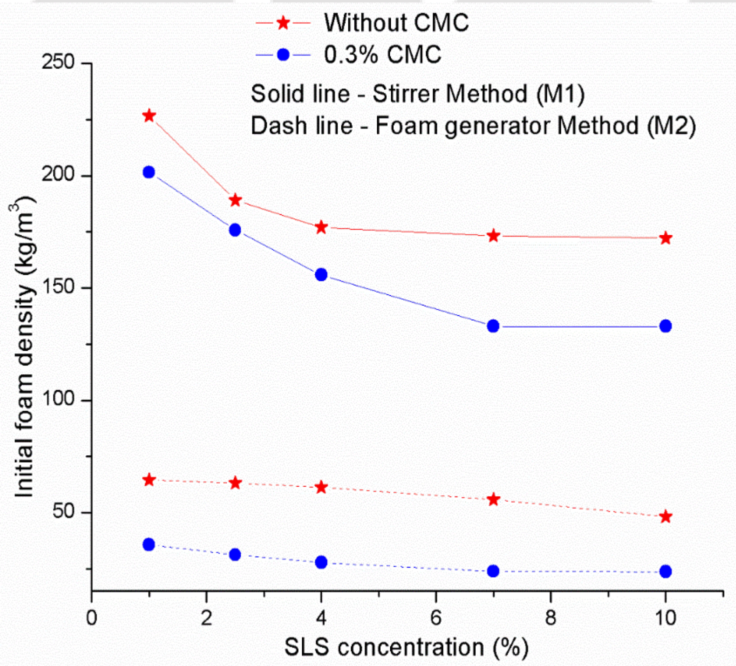


Fig. 3.12 Comparison of initial foam density of foam produced with and without CMC for methods M1 and M2 (FGP 490 kPa)

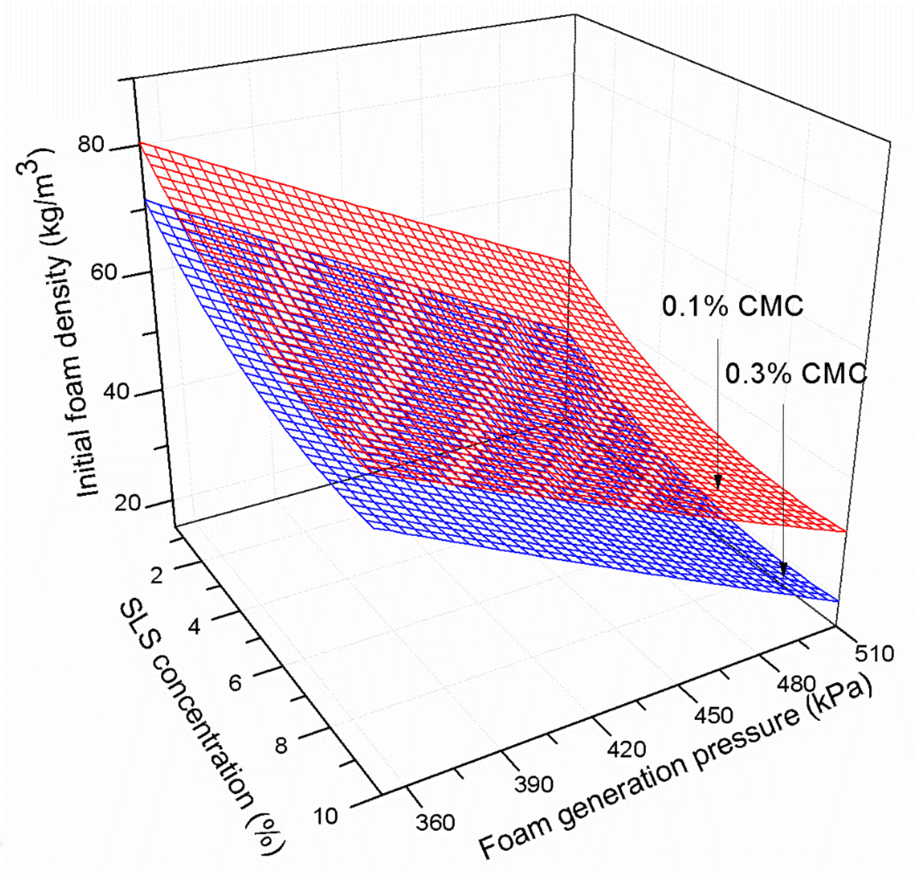


Fig. 3.13 Response surface plots for initial foam density for M2 (CMC 0.1% and 0.3%)

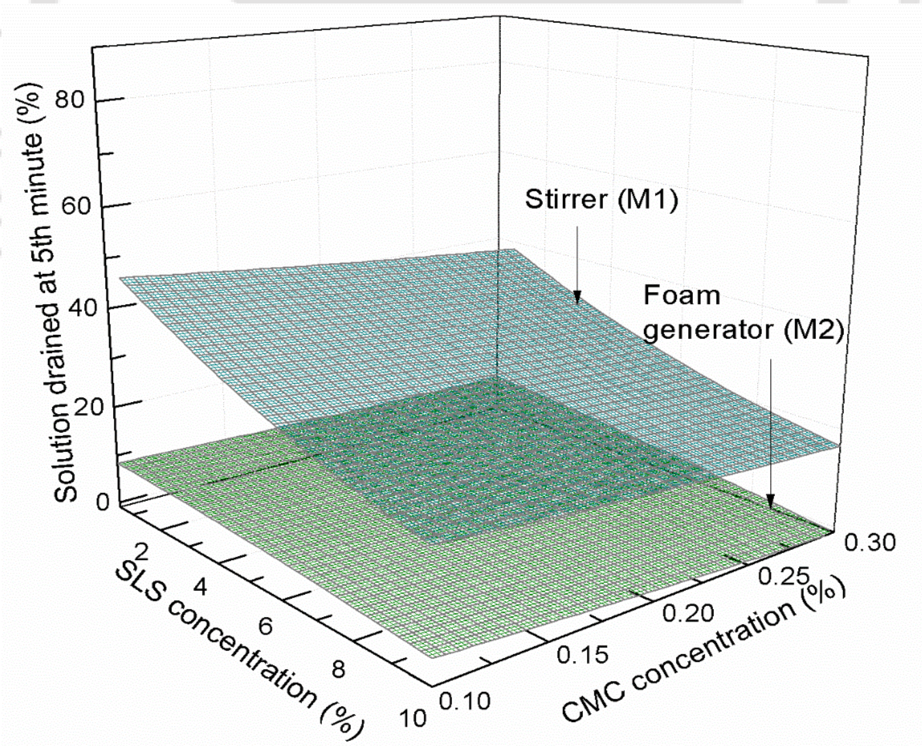
3.3.4 Response Surface Model for Foam Stability

Fig. 3.14 shows the response surface plots for percentage solution drained for foam produced using method M1 and M2 at 5, 10 and 15 minutes after foam generation. Foam produced using stirrer being wet foam evidently shows higher drainage. Percentage solution drained of foam produced using both the methods reduces significantly with increase in SLS and CMC concentration. The reduction in percentage solution drained or improvement in stability of foam can be ascribed to the production of good quality of foam (with lesser liquid fraction) at higher levels of SLS and CMC concentration. In particular, the addition of CMC results in enhancement of viscosity of surfactant solution and production of stable foam with smaller size bubble microstructure as explained in upcoming sections.

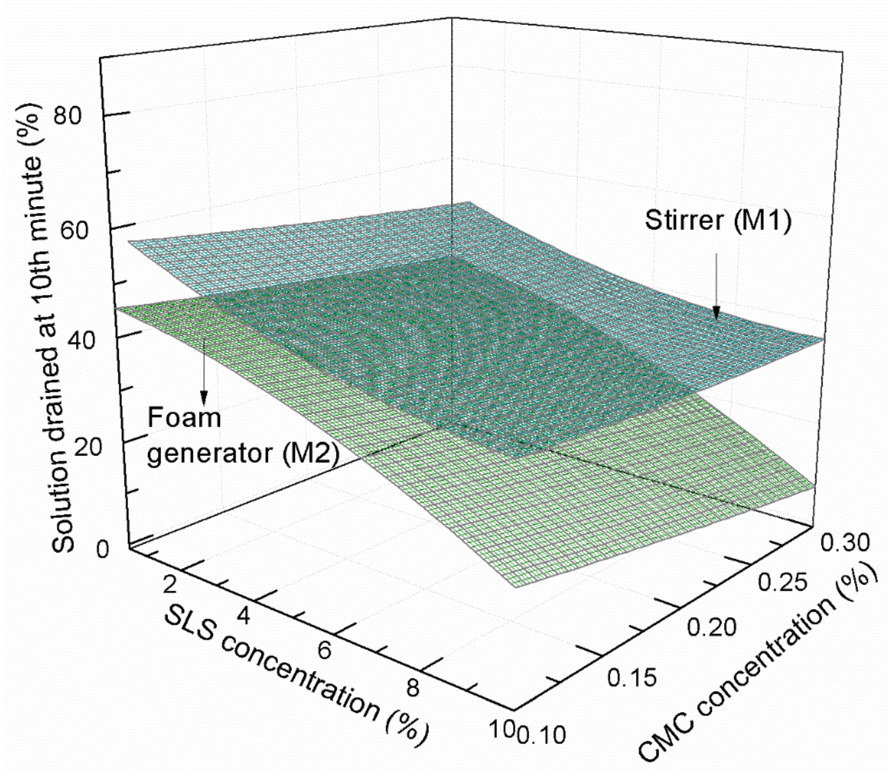
Furthermore the reduction in solution drained due to addition of CMC is more predominant for foam produced using method M2 which could be again attributed to efficient role of CMC in foam produced based on compressed air method when compared to that of stirrer. For instance, a reduction of 76% in drainage is observed for foam produced with 0.3%

CMC when compared to foam without CMC at 1% SLS concentration for generator method M2 (Fig. 3.15). Also the early rate of drainage in the first five minutes is very low for foam produced using method M2 compared to that of M1. However, the later drainage values at 15th minute is higher for foam based on M2 particularly at very low values of SLS and CMC concentration. In addition it is to be noted that the effect of variation of SLS and CMC concentration is more significant on foam drainage when compared to that of initial foam density.

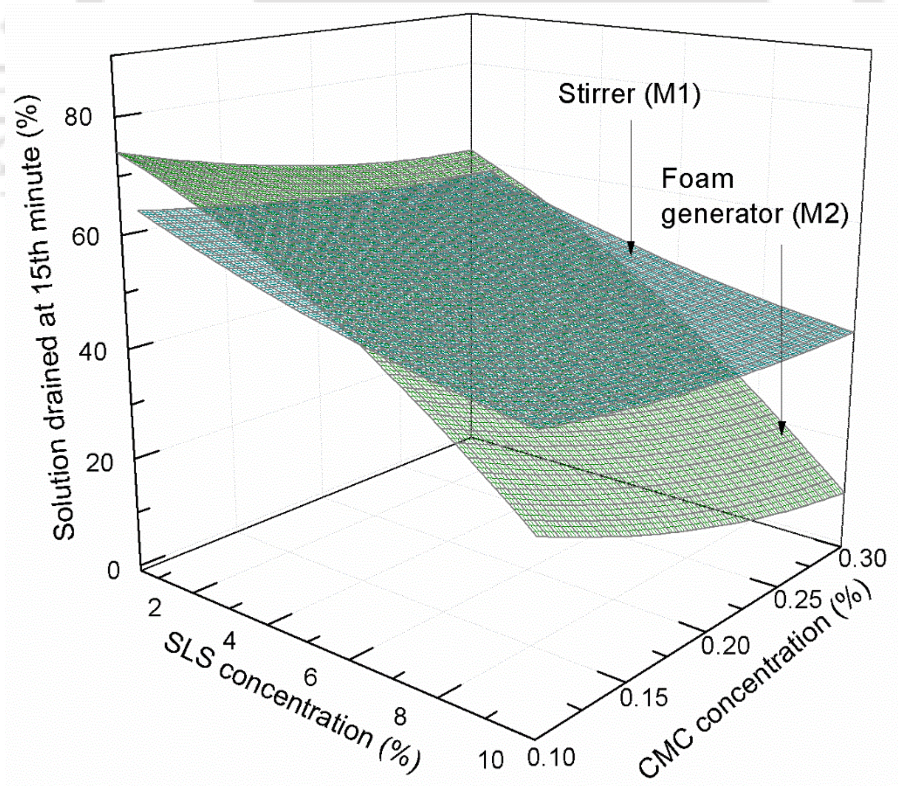
Fig. 3.16 shows the variation of percentage solution drained with SLS concentration and foam generation pressure at different levels of CMC concentration. Similar to earlier observations on the effect of FGP on IFD, the foam drainage reduces significantly with increase in FGP. Hence again from stability point of view, it is preferable to choose higher SLS, CMC concentrations and higher FGP for the foam generator used. However it is observed that adoption of FGP beyond 490 kPa results in interrupted foam production affecting the foam output rate. Hence for the current foam generator model the recommended pressure is restricted to 490 kPa.



(a) 5th minute



(b) 10th minute



(c) 15th minute

Fig. 3.14 Response surface plots for percentage solution drained for M1 and M2 (FGP 490 kPa)

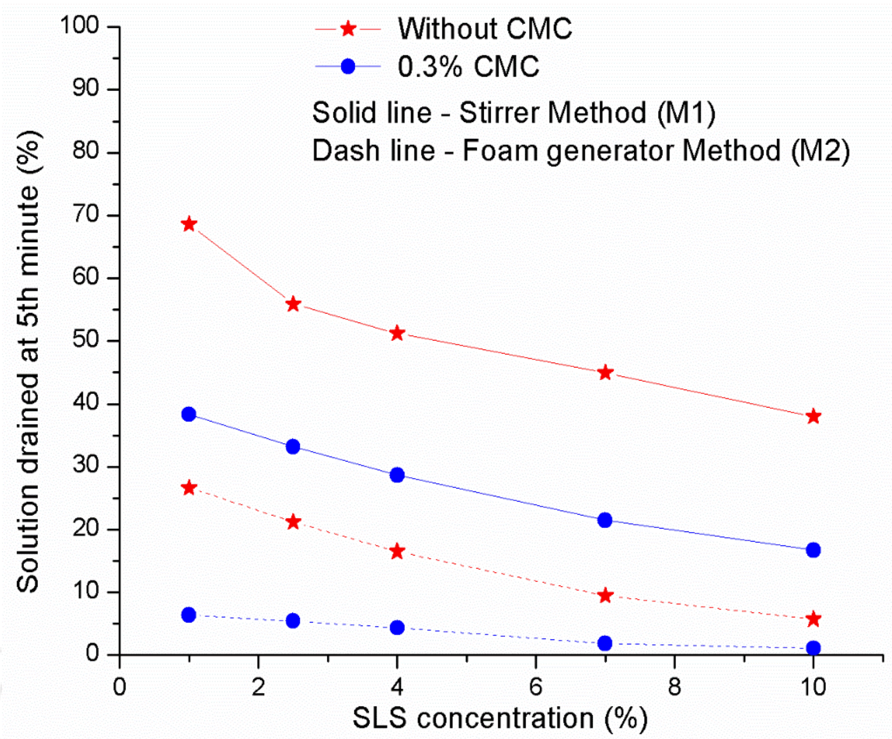


Fig. 3.15 Comparison of percentage solution drainage of foam produced with and without CMC for method M1 and M2 (FGP 490 kPa)

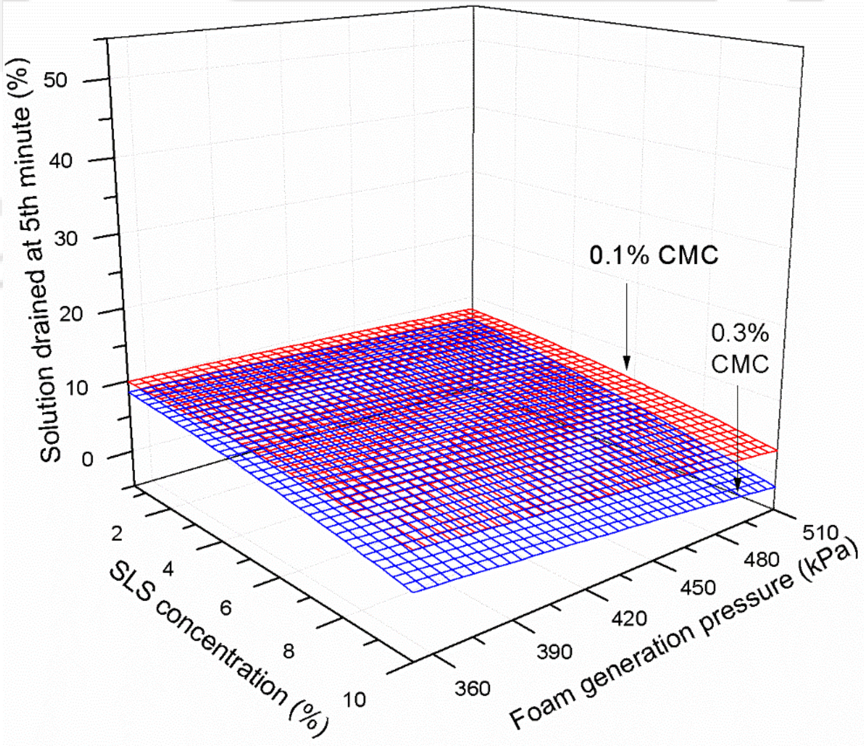


Fig. 3.16 Response surface plots for percentage solution drained at 5th minute for M2 (CMC 0.1% and 0.3%)

3.3.5 Response Surface Model for Viscosity of Surfactant Solution

The response surface plot for viscosity of surfactant solution is plotted in Fig. 3.17. It is noticed from Fig. 3.17 that the viscosity of surfactant solution at low levels of SLS and CMC concentration is very less and this gets enhanced significantly with increase in SLS and CMC concentration. Furthermore results indicates that the significant increase in viscosity of SLS surfactant solution is achieved only in presence of CMC (Fig. 3.18). This could be attributed to the tight packing of surfactant molecules achieved due to the addition of CMC which thereby enhances the viscosity of surfactant solution significantly (Sharma and Sharma 1991). Also as established earlier, that the viscous surfactant solution can result in stable foam with smaller bubble microstructure (Bonsu et al. 2016) and hence, it is preferable to adopt higher SLS and CMC concentration. However as the viscosity of surfactant solution can also affect the workability of foam concrete mix, the optimization of CMC dosage must also consider the workability and stability of foam concrete mix as discussed in chapter 5.

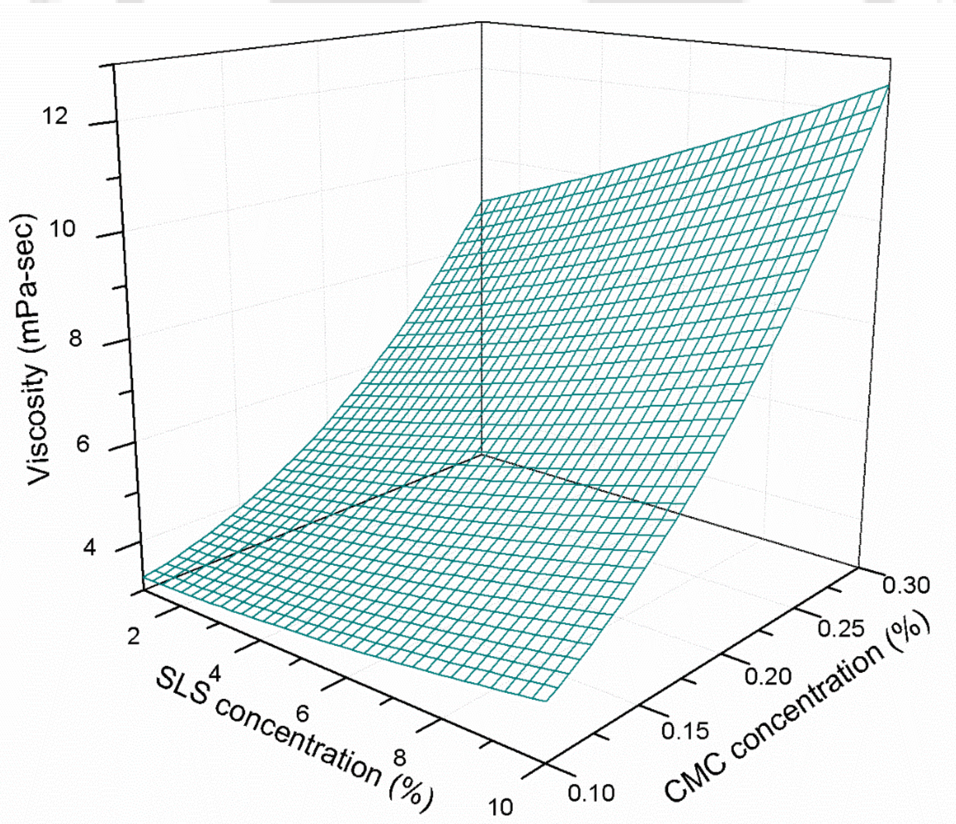


Fig. 3.17 Response surface plot for viscosity of surfactant solution

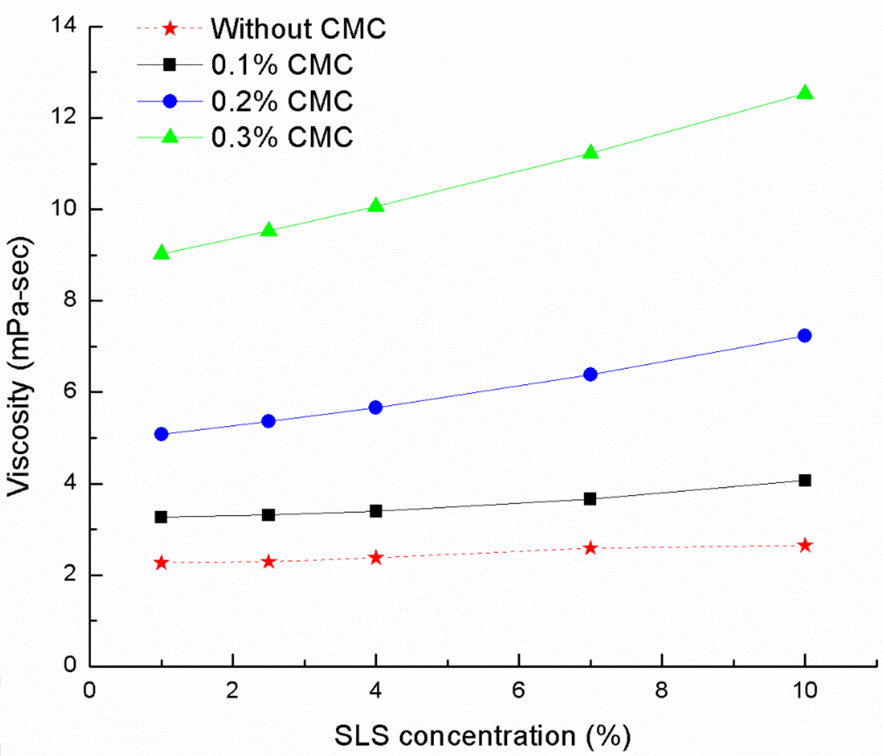


Fig. 3.18 Comparison of viscosity of surfactant solution with and without CMC

3.3.6 Response Surface Model for Average Bubble Diameter of Foam

Fig. 3.19 shows the response surface plot for variation in average bubble diameter of foam produced using methods M1 and M2. It is observed for both methods M1 and M2 that the adoption of higher SLS and CMC concentration results in foam with reduced bubble diameter. For instance, a reduction of 49% in bubble size is observed for foam produced with 0.3% CMC when compared to that of foam without CMC at lower SLS concentration (1%) for generator method M2 (Fig. 3.20). The reduction in bubble size can be attributed to the increase in viscosity of surfactant solution due to addition of CMC as explained in earlier section. Hence the above results are in agreement with the earlier observations by other researchers (Kroezen et al. 1988) that highly viscous surfactant solution can produce foam with smaller bubble microstructure (Bonsu et al. 2016).

Comparison of bubble sizes of foam shows that foam which is basically a wet foam (with liquid fraction ranging from 13 to 23%) has relatively finer bubble structure compared to dry foam (with liquid fraction from 1.9 to 9.4%) particularly when lower surfactant and CMC concentration is adopted (Fig. 3.19). This is in line with earlier observations by Bonsu et al. (2016) who have established that dispersion of more volume of air per unit volume of surfactant solution in dry foam can be the reason for coarser bubble structure in dry foam with lesser liquid fraction. However, use of higher surfactant and CMC concentration, results in production of more viscous surfactant solution eventually resulting in smaller size bubble microstructure (Fig. 3.19 and Fig. 3.20). Hence the influence of method of foam production (M1 and M2) on bubble size is less significant on foam produced at higher surfactant and CMC concentration wherein viscosity of surfactant solution plays a major role. Fig. 3.21 shows the variation of average bubble diameter with FGP at different levels of CMC concentration for method M2. A reduction of 30% in average bubble diameter is observed for variation in FGP from 353 to 490 kPa at higher levels of SLS (10%) and CMC concentration (0.3%).

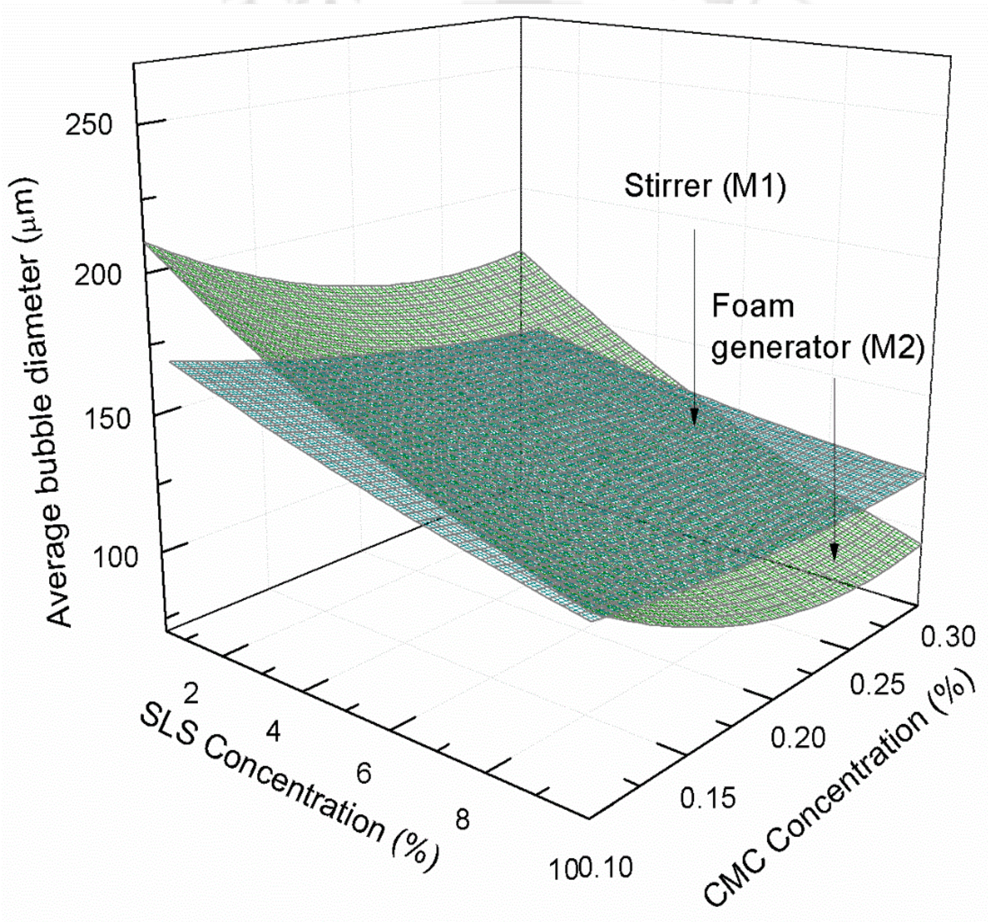


Fig. 3.19 Response surface plots for average bubble diameter for M1 and M2

(FGP 490 kPa)

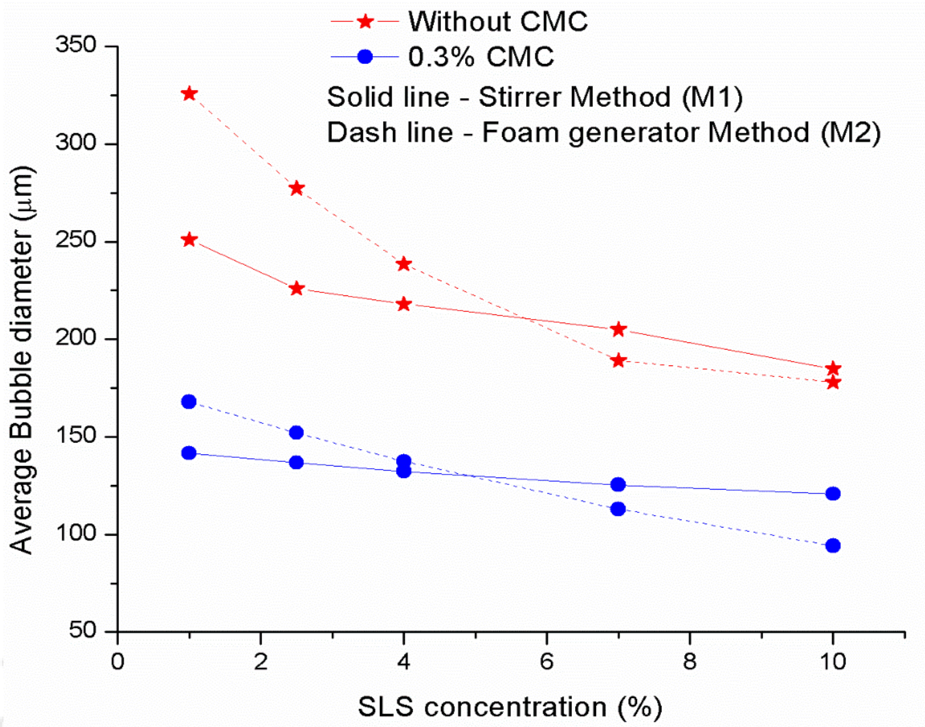


Fig. 3.20 Comparison of average bubble diameter for foam produced with and without CMC for method M1 and M2 (FGP 490 kPa)

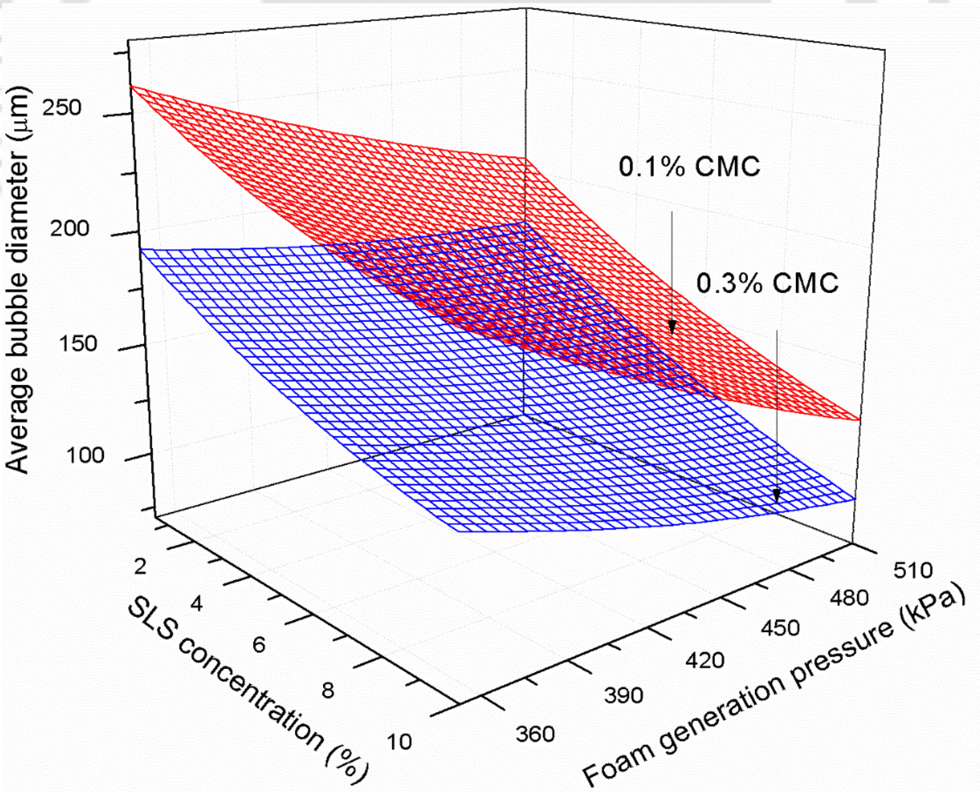


Fig. 3.21 Response surface plots for average bubble diameter for M2 (CMC 0.1% and 0.3%)

3.3.7 Relation between Initial Foam Density and Viscosity of Surfactant Solution

A scatter plot showing the relation between viscosity of surfactant solution and initial foam density of foam produced with methods M1 and M2 are plotted in Fig. 3.22. It is observed that an increase in surfactant concentration (at a constant CMC concentration) associated with some increase in viscosity results in significant reduction of the initial foam density up to SLS concentration of 7% after which there is not much variation for foam produced with both methods M1 and M2. For instance, 33% reduction in initial foam density is observed due to increase in SLS concentration from 1 to 7%, at a constant CMC concentration of 0.2% for foam produced using method M1. This trend of reduction in initial foam density with increase in SLS concentration could be attributed to the significant improvement in quality of foam due to increase in surfactant concentration. On the other hand, the significant increase in viscosity of surfactant solution achieved due to increase in CMC concentration (at a constant SLS concentration) does not result in much variation in initial foam density (% reduction in IFD less than 4%) for method M1.

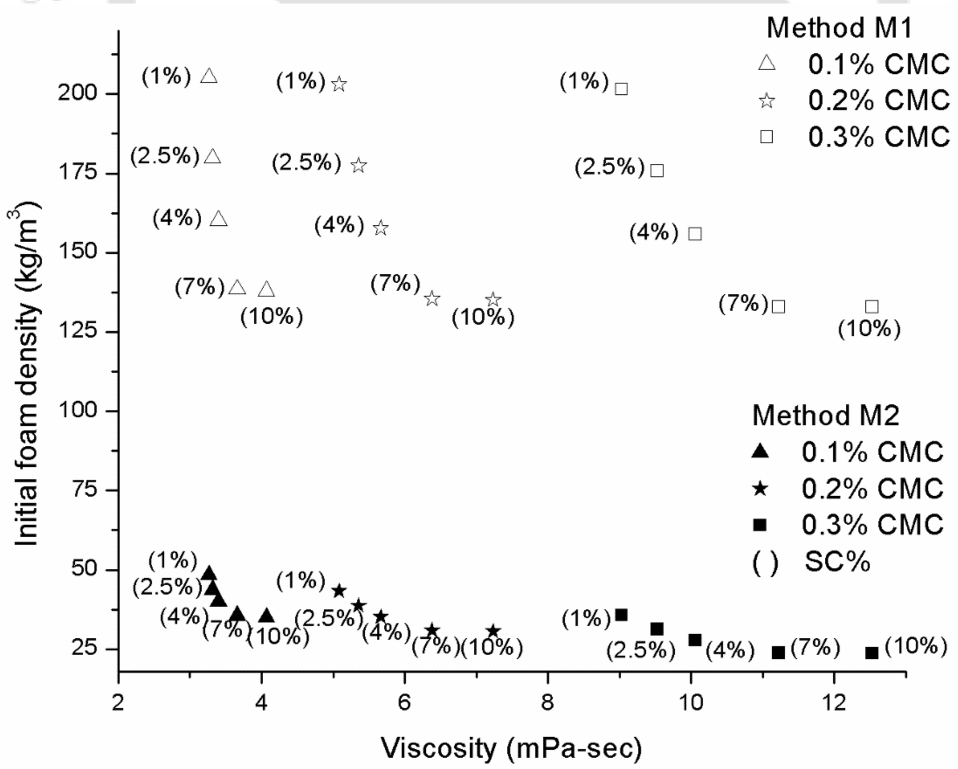


Fig. 3.22 Variation of initial foam density with viscosity of surfactant solution

However for method M2, significant increase of 208 % in viscosity achieved due to increase in CMC concentration from 0.1 to 0.3% at constant SLS concentration of 10%

results in 32% reduction in initial foam density. Hence based on the above observations it can be concluded that the influence of increase in viscosity achieved due to addition of the CMC on improvement of quality of foam (reduction of liquid fraction) is realized to a greater extent in foam produced using foam generator based on compressed air method.

3.3.8 Relation between Foam Stability and Viscosity of Surfactant Solution

The relation between viscosity and percentage solution drained at 5th minute after foam generation for foam produced with stirrer (M1) and generator (M2) are plotted in Fig. 3.23.

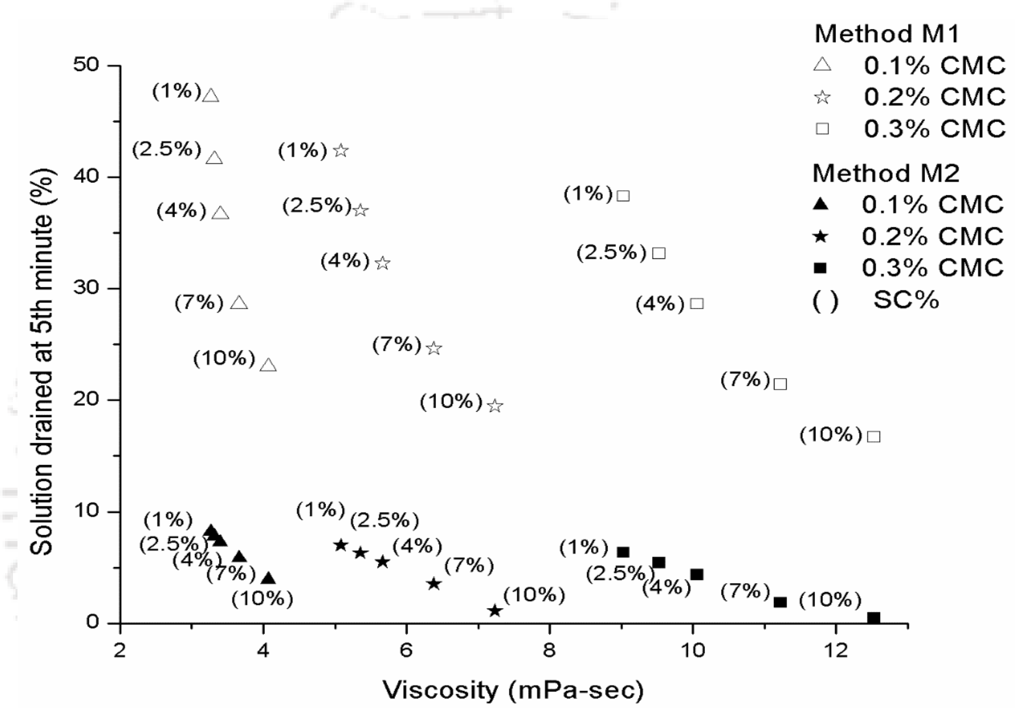


Fig. 3.23 Variation of solution drained with viscosity of surfactant solution

Similar to discussion on foam density, it is observed that an increase in surfactant concentration (at a constant CMC concentration) associated with some increase in viscosity results in significant reduction in percentage solution drained for both M1 and M2. For instance, 54% reduction in solution drained is observed due to increase in SLS concentration from 1 to 10%, at a constant CMC concentration of 0.2% for foam produced using method M1. Furthermore the significant increase in viscosity of surfactant solution achieved due to increase in CMC concentration (for a constant SLS concentration) results in significant reduction in foam drainage. It can be observed apparently for foam produced with M2, that the percentage solution drained reduces significantly to less than 1% particularly at higher concentration of CMC which confirms the earlier observation that role of CMC in foam quality improvement is more efficient in compressed air method of foam production. The reason for reduction in drainage due to increase in viscosity of surfactant solution can be ascribed to the cushioning effect of viscous lamellae around the bubble which in turn retards the foam drainage. Also, the smaller size bubble microstructure produced with higher viscous surfactant solution can also contribute to improve stability as explained in the earlier section. Adding to above, it has been reported by (Kroezen et al. 1988) that the smaller bubble microstructure of foam needs more stress for deformation and hence it results in improved stability.

Furthermore drier foam produced with method M2 has very less solution drained at 5th minute compared to M1 due to its lesser liquid fraction. Also it is to be noted that even though wet foam (M1) has finer bubble structure, because of its higher liquid fraction, its drainage is very high compared to that of M2 proving that, apart from bubble size, the amount of liquid fraction also plays a significant role in drainage of foam.

3.4 SUMMARY

A systematic study on variation of important foam characteristics such as initial foam density, foam stability, bubble size and viscosity of surfactant solution with foam production parameters are carried out through experiment design based on Response surface methodology for two different foam production methods namely stirrer (M1) and foam generator (M2). The possible response surface models have been developed for predicting the variation of foam characteristics as the function of foam production parameters. Studies on relative foam behaviour indicates, that the adoption of higher levels of SLS and CMC concentration are recommended for both methods of production to produce highly viscous surfactant solution which can result in stable foam with lesser liquid fraction and smaller bubble size microstructure. Furthermore there is a strong relationship between viscosity of surfactant solution, initial foam density, foam stability and bubble microstructure. The comparative studies on foam production methods has established that foam produced based on compressed air method (foam generator) is of better quality with lesser liquid fraction and drainage compared to that of foam produced with stirrer. Also the potential of CMC in improvement of quality of foam (reduction of liquid fraction) is utilized to a greater extent in foam produced using foam generator based on compressed air method. Hence the further studies are carried out with compressed air method of foam production.

Chapter 4

RELATIVE PERFORMANCE EVALUATION OF FOAM PRODUCED WITH TWO SYNTHETIC SURFACTANTS (SODIUM LAURYL SULPHATE AND NONYL PHENOL ETHOXYLATED) AND ADDITIVE (CARBOXYMETHYL CELLULOSE)

4.1 GENERAL

The chapter 3 comprises of evaluation of influence of foam production method on the properties of foam produced with surfactant sodium lauryl sulphate (SLS). The compressed air method of production of foam is found to result in relatively drier and stable foam suitable for foam concrete production. Hence as a next step, the relative behaviour of foam produced with two different synthetic surfactants sodium lauryl sulphate (SLS) and nonyl phenol ethoxylated (NPE) using compressed air method of production is studied through a systematic experimental design based on Response surface methodology. Furthermore its suitability for use in foam concrete production is assessed as per guidelines of ASTM C-796-97.

4.2 MATERIALS AND METHODS

Foam is produced by aerating two commercially available synthetic surfactants viz., sodium lauryl sulphate and nonyl phenol ethoxylated with additive carboxymethyl cellulose using compressed method of foam production with foam generator. Preliminary trials with NPE, reveals that at least 3% surfactant concentration is needed to produce foam with foam generator. Hence based on preliminary studies, the factor levels are designed for NPE as shown in Table 4.1. As explained in chapter 3 for surfactant SLS, in a similar fashion the experimental design matrix (Table 4.2) and data analysis are performed for NPE using Design Expert software.

Table 4.1 Factors and factor levels adopted for design matrix (NPE)

Notation	Factor	Low level	High level
NPE	NPE Concentration (%)	3	10
CMC	CMC Concentration (%)	0.1	0.3
FGP	Foam generation pressure (kPa)	431	588

Table 4.2 Matrix of the central composite design (NPE)

Treatment runs	NPE concentration (%)	CMC concentration (%)	Foam generation pressure (kPa)
1	4.41	0.25	556.59
2	10	0.2	510
3	6.5	0.2	588
4	8.58	0.25	556.59
5	6.5 (C)	0.2	510
6	6.5	0.1	510
7	8.58	0.25	463.29
8	6.5 (C)	0.2	510
9	6.5 (C)	0.2	510
10	6.5	0.2	431
11	6.5 (C)	0.2	510
12	6.5	0.3	510
13	4.41	0.14	463.29
14	8.58	0.14	463.29
15	3	0.2	510
16	6.5 (C)	0.2	510
17	8.58	0.14	556.59
18	4.41	0.25	463.29
19	6.5 (C)	0.2	510
20	4.41	0.14	556.59

C: Centre point

4.3 Regression Analysis

The details of model equations for initial foam density, foam stability (foam drainage at various time intervals after foam generation), viscosity of surfactant solution and average bubble diameter of foam for NPE are presented in Table 4.3. Experimental validation for second order polynomial regression models are observed to be highly adequate with R^2 (> 0.95) for most of the regression models (Table 4.4 and Table 4.5).

Table 4.3 RSM for different properties of foam and surfactant (NPE)

Foam Properties	Response surface models
Initial foam density (kg/m^3)	+114.837 + (3.619*NPE) + (328.445*CMC) - (23.410*FGP) + (5.483*NPE*CMC) - (1.147*NPE*FGP) - (33.229*CMC*FGP) - (0.031*NPE^2) - (299.618*CMC^2) + (2.250*FGP^2)
Foam drainage at 5^{th} minute (%)	+32.064 - (1.654*NPE) - (61.330*CMC) - (6.666*FGP) + (1.031*NPE*CMC) + (0.176*NPE*FGP) + (7.073*CMC*FGP) + (0.025*NPE^2) + (32.620*CMC^2) + (0.330*FGP^2)
Foam drainage at 10^{th} minute (%)	+307.570 - (6.346*NPE) - (448.511*CMC) - (71.391*FGP) - (2.294*NPE*CMC) + (0.462*NPE*FGP) + (47.504*CMC*FGP) + (0.141*NPE^2) + (390.302*CMC^2) + (4.716*FGP^2)
Foam drainage at 15^{th} minute (%)	+269.59 - (6.883*NPE) - (480.221*CMC) - (40.573*FGP) + (0.485*NPE*CMC) + (0.915*NPE*FGP) + (66.408*CMC*FGP) - (0.140*NPE^2) + (120.782*CMC^2) + (0.836*FGP^2)
Viscosity of surfactant solution (mPa-sec)	+8.09+(1.092*NPE)-(73.77*CMC)+(5.571*NPE*CMC)-(0.042*NPE^2)+(333.124*CMC^2)
Average bubble diameter (μm)	+628.666 - (42.817*NPE) - (152.904*CMC) - (93.814*FGP) - (14.142*NPE*CMC) - (1.767*NPE*FGP) - (79.549*CMC*FGP) + (3.029*NPE^2) + (1161.328*CMC^2) + (10.333*FGP^2)

Similar to the observations on SLS, the normal plot of residuals confirms the normality assumptions and the residual plots displays the constant variance as shown in (Fig. 4.1 to Fig. 4.6). Furthermore the additional tests are also conducted to confirm the accuracy of prediction relations and the test results shows that the difference between the predicted and observed responses is within 10% as presented in Table 4.6.

Table 4.4 ANOVA and regression coefficients of response surface models fitted (NPE)

Variables		Main effects			Quadratic effects			Interaction effect		
		x_1	x_2	x_3	${x_1}^2$	${x_2}^2$	${x_3}^2$	$x_1\ x_2$	$x_1\ x_3$	$x_2\ x_3$
Initial foam density (kg/m^3)	p-value	0.0043	0.0011	< 0.0001	0.8834	0.2744	0.5905	0.5932	0.3775	0.4623
	F value	13.51	20.38	50.84	0.023	1.34	0.31	0.30	0.85	0.58
Foam drainage at 5th minute(%)	p-value	< 0.0001	< 0.0001	< 0.0001	0.0076	0.0065	0.0504	0.0180	0.0031	0.0013
	F value	137.51	67.94	83.94	11.08	11.74	4.94	7.98	14.94	19.61
Foam drainage at 10th minute(%)	p-value	< 0.0001	0.0001	< 0.0001	0.3152	0.0383	0.0950	0.7223	0.5689	0.1144
	F value	80.58	36.35	61.96	1.12	5.68	3.40	0.13	0.35	2.99
Foam drainage at 15th minute(%)	p-value	< 0.0001	< 0.0001	< 0.0001	0.2730	0.4347	0.7258	0.9337	0.2270	0.0236
	F value	221.20	84.72	124.46	1.35	0.66	0.13	$7.268*10^{-003}$	1.66	7.11
Viscosity (mPa-sec)	p-value	< 0.0001	< 0.0001	-	0.0402	< 0.0001	-	0.0002	-	-
	F value	1767.96	4847.60	-	6.31	255.42	-	50.32	-	-
Average bubble diameter (µm)	p-value	<0.0001	<0.0001	0.0008	<0.0001	0.0127	0.1150	0.3585	0.3585	0.2441
	F value	534.11	68.69	22.09	93.84	9.19	2.98	0.93	0.93	1.53

Table 4.5 R^2, Probability value and F values for the final models (NPE)

Response variable	R^2	R^2 adj	Regression p value	F value
Initial foam density (kg/m^3)	0.8982	0.8067	0.0007	9.81
Foam drainage at 5th minute (%)	0.9726	0.9480	< 0.0001	39.48
Foam drainage at 10th minute (%)	0.9503	0.9055	< 0.0001	21.24
Foam drainage at 15th minute (%)	0.9779	0.9579	< 0.0001	49.06
Viscosity (mPa-sec)	0.9990	0.9983	< 0.0001	1388.56
Average bubble diameter (µm)	0.9864	0.9742	<0.0001	80.75

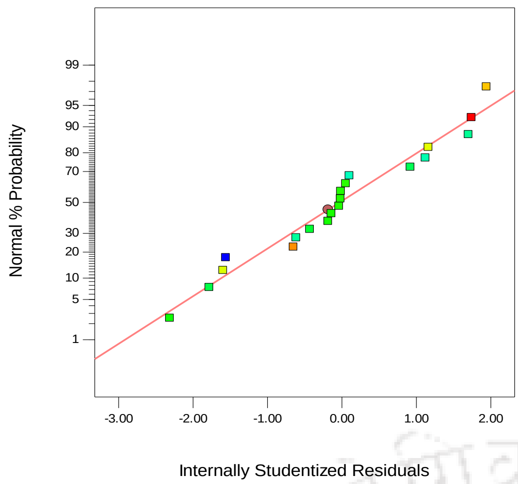


Fig 4.1 (a) Normal probability plot (NPE)

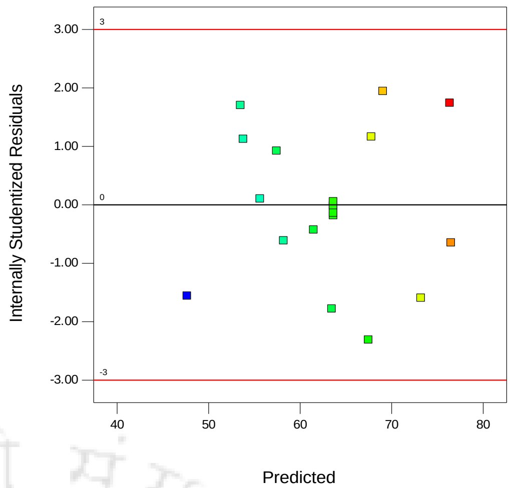


Fig 4.1 (b) Residual plot (NPE)

Fig. 4.1 Diagnostic plots for initial foam density (NPE)

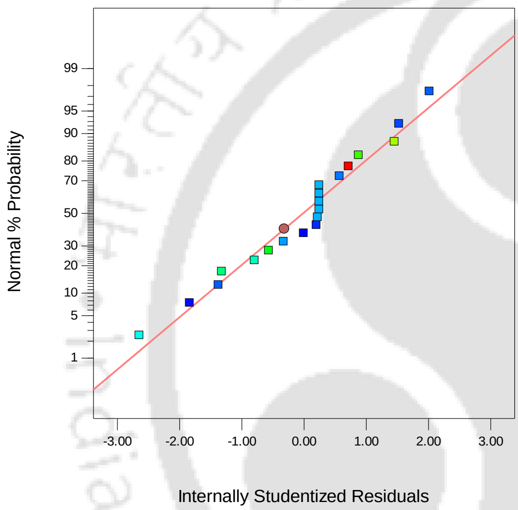


Fig 4.2 (a) Normal probability plot (NPE)

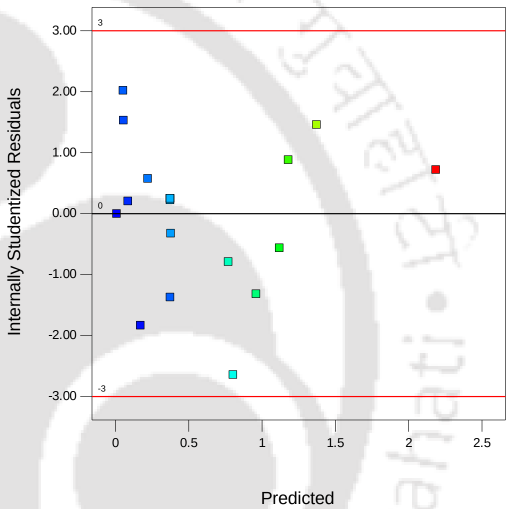


Fig 4.2 (b) Residual plot (NPE)

Fig. 4.2 Diagnostic plots for % solution drained at 5^{th} minute after foam generation (NPE)

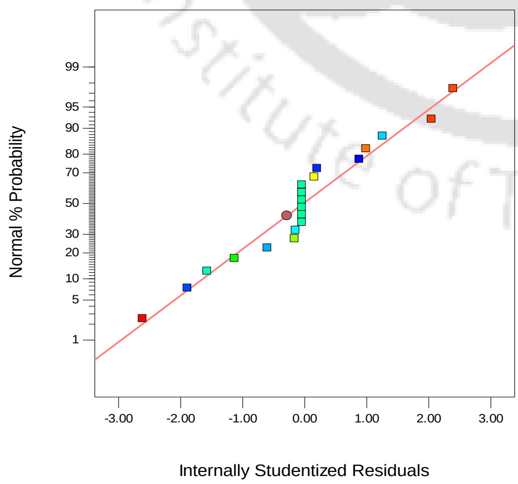


Fig 4.3 (a) Normal probability plot (NPE)

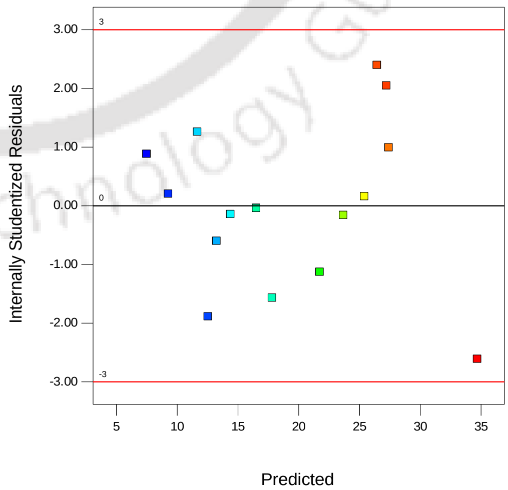


Fig 4.3 (b) Residual plot (NPE)

Fig. 4.3 Diagnostic plots for % solution drained at 10^{th} minute after foam generation (NPE)

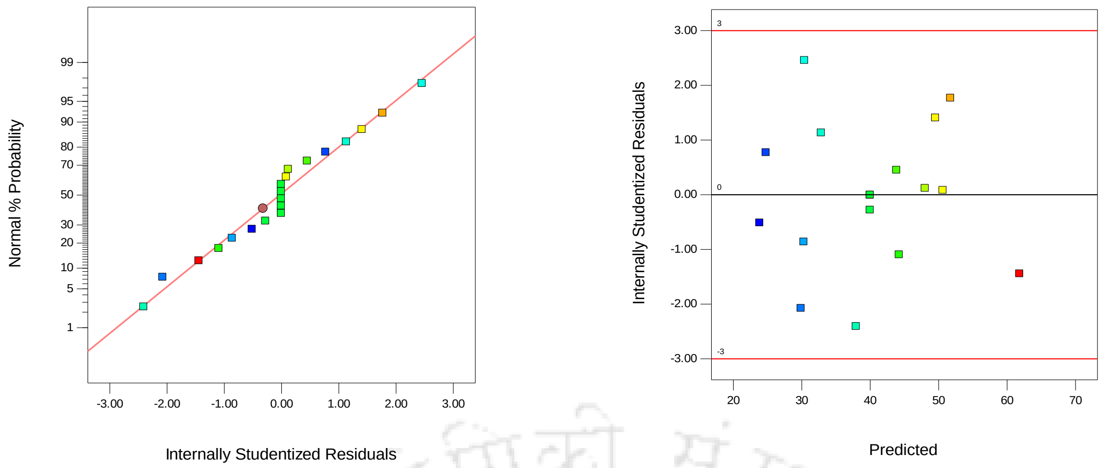


Fig 4.4 (a) Normal probability plot (NPE) Fig 4.4 (b) Residual plot (NPE)

Fig. 4.4 Diagnostic plots for % solution drained at 15th minute after foam generation (NPE)

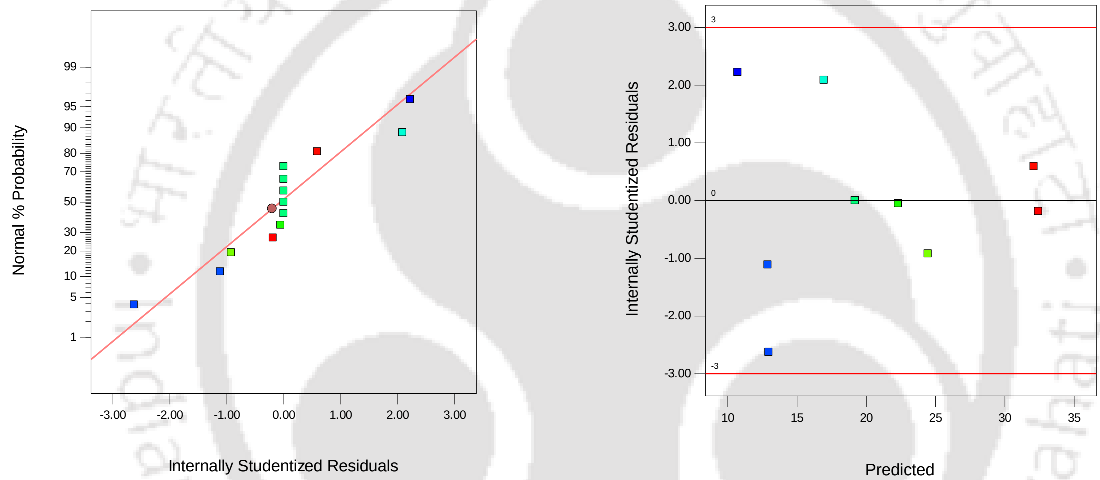


Fig 4.5 (a) Normal probability plot (NPE) Fig 4.5 (b) Residual plot (NPE)

Fig. 4.5 Diagnostic plots for viscosity of surfactant solution (NPE)

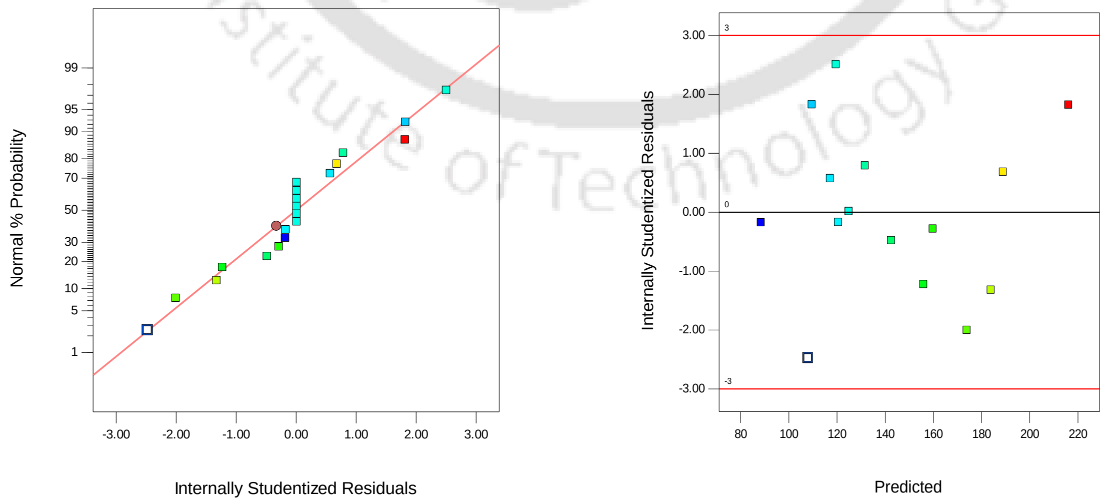


Fig 4.6 (a) Normal probability plot (NPE) Fig 4.6 (b) Residual plot (NPE)

Fig. 4.6 Diagnostic plots for average bubble diameter of foam (NPE)

Table 4.6 Observed and predicted responses for confirmation of models (NPE)

Sl	Factors			Initial foam density (kg/m^3)			Solution drained at 5th minute (%)			Solution drained at 10th minute (%)			Solution drained at 15th minute (%)			Viscosity (mPa-sec)			Average bubble diameter (μm)		
	NPE (%)	CMC (%)	FGP (kPa)	P	O	D	P	O	D	P	O	D	P	O	D	P	O	D	P	O	D
1	3	0.1	490	62.29	64.31	-3.24	2.95	2.71	8.14	35.88	33.41	6.88	84	76.12	9.38	8.60	8.36	2.79	242.53	245.64	-1.28
2	4.4	0.2	549	62.68	56.99	9.08	0.53	0.49	7.55	18.19	16.54	9.07	47.32	50.03	-5.73	15.54	14.34	7.72	168.38	165.52	1.70
3	9.5	0.18	490	59.96	59.91	0.08	0.15	0.16	-6.67	16.79	15.87	5.48	31.95	30.65	4.07	21.65	20.46	5.49	114.94	120.22	-4.59
4	6.2	0.13	490	60.16	65.16	-8.31	1.2	1.29	-7.50	26.02	27.44	-5.46	54.24	56.06	-3.36	13.75	14.15	-2.90	150.76	155.44	-3.10
5	5.5	0.2	510	65.27	63.61	2.54	0.59	0.57	3.39	17.66	18.2	-3.06	43.79	45.4	-3.68	17.50	18.2	-4.00	143.42	151.91	-5.92
6	3	0.3	510	71.3	78.33	-9.86	0.85	0.78	8.24	10.68	10.16	4.87	27.95	26.82	4.04	23.84	22.2	6.87	213.3	207.54	2.70
7	7	0.14	470	62.71	62.62	0.14	1.11	1.12	-0.90	24.67	25.84	-4.74	49.69	53.61	-7.89	15.30	14.3	6.53	139.81	148.4	-6.14

P – Predicted Value

O – Observed Value

D – % Difference = [(Predicted – Observed)/ Predicted]*100

4.3.1 Response Surface Models for Initial Foam Density

Similar to behaviour of SLS as discussed earlier, initial foam density of NPE reduces with increase in surfactant concentration due to improvement in quality of foam (reduction in liquid fraction). However, the variation of initial foam density with surfactant concentration at 0.3% CMC concentration is not significant for NPE which can be attributed to the very high viscous nature of NPE foam produced at high level of CMC concentration. Furthermore, the increase in CMC concentration for a given NPE concentration results in significant increase in initial foam density unlike SLS foam where decreasing trend is observed (Fig. 4.7). For instance an increase in CMC concentration from 0% to 0.3% results in 47% enhancement of initial foam density at a NPE concentration of 10% (Fig. 4.8). The reason for the difference in behaviour between the foam produced with these two surfactants can be attributed to the very high viscosity of NPE surfactant solution particularly at higher level of CMC concentration. Hence CMC is found to play a more efficient role in viscosity enhancement with non-ionic surfactant NPE when compared to that of anionic surfactant SLS. Furthermore a similar kind of relationship between foam density and viscosity has been demonstrated by Siva et al. (2015). Fig. 4.9 shows that an increase in foam generation pressure results in significant reduction of initial foam density of NPE foam similar to the observations on SLS foam.

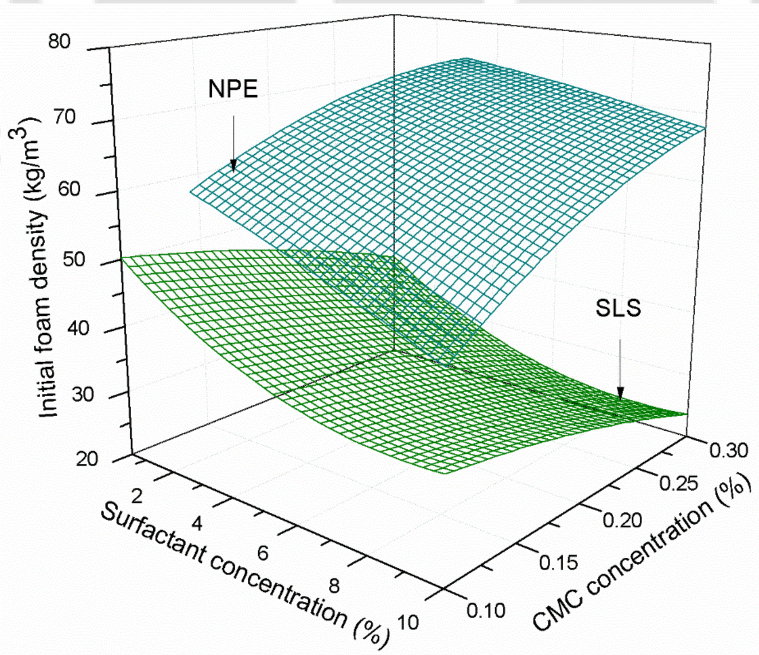


Fig. 4.7 Response surface plots for initial foam density for SLS and NPE (FGP 490 kPa)

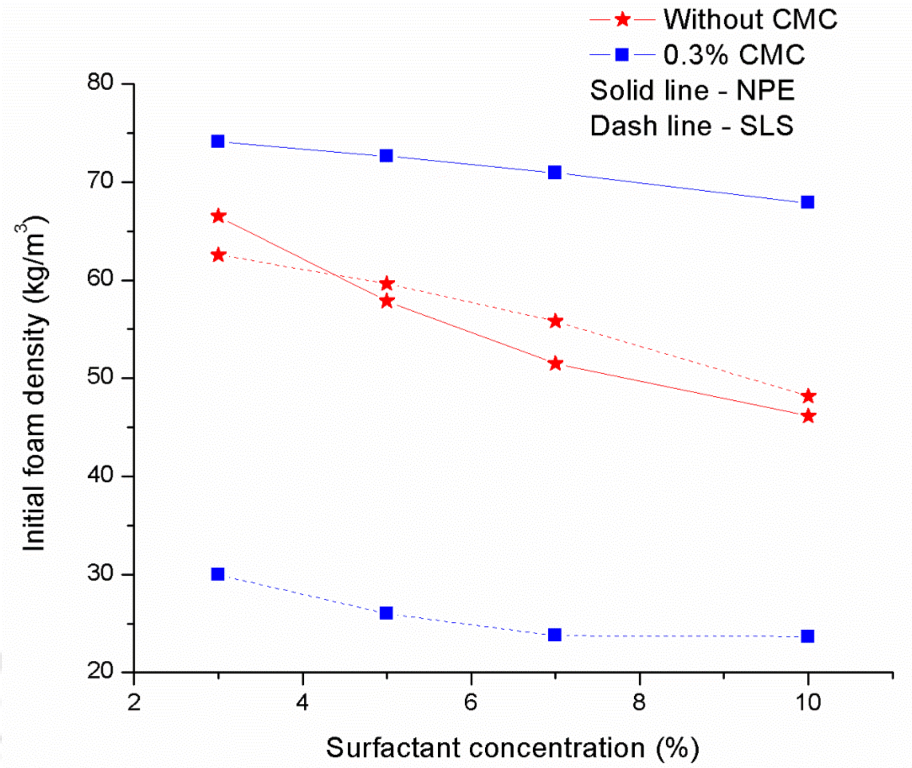


Fig. 4.8 Comparison of initial foam density of foam produced with and without CMC for surfactants SLS and NPE

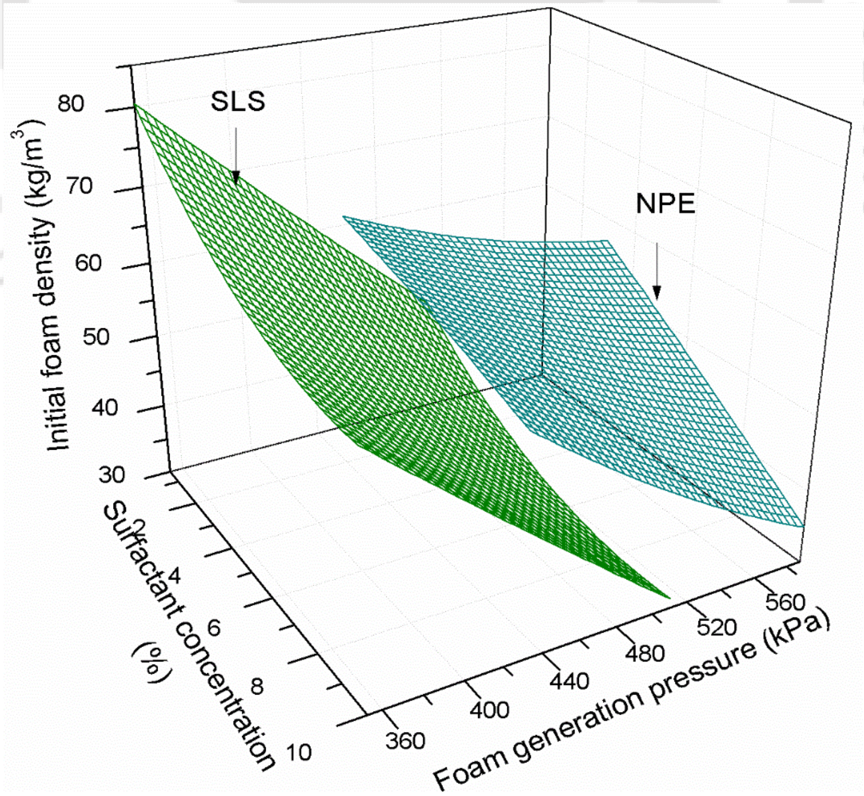
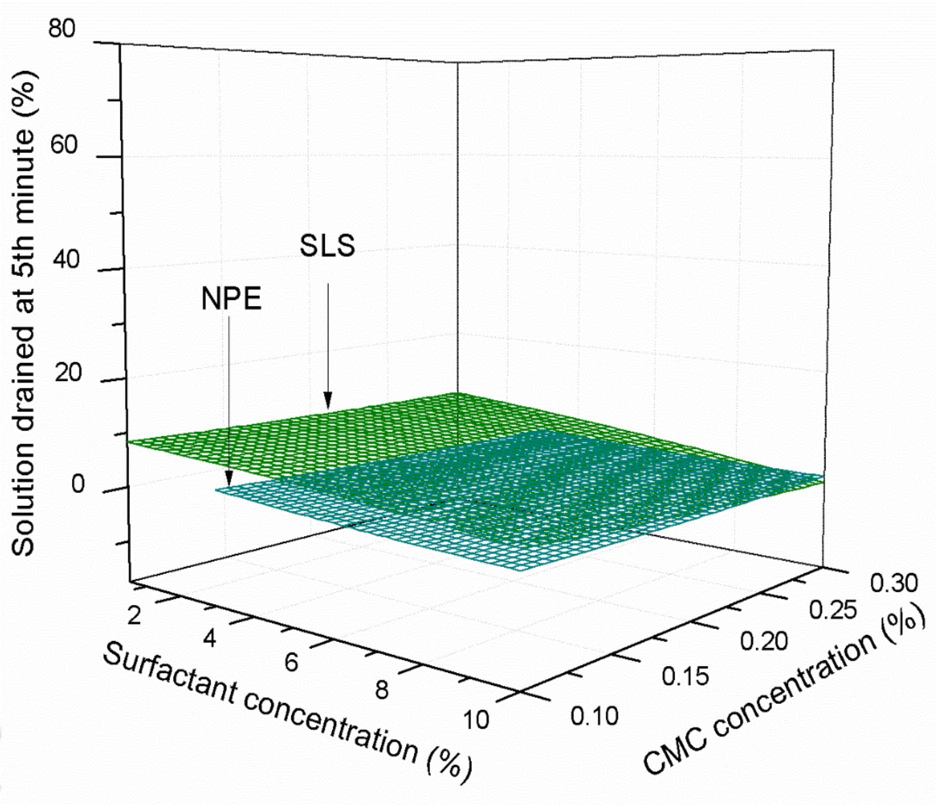


Fig. 4.9 Response surface plots for initial foam density for SLS and NPE (CMC 0.1%)

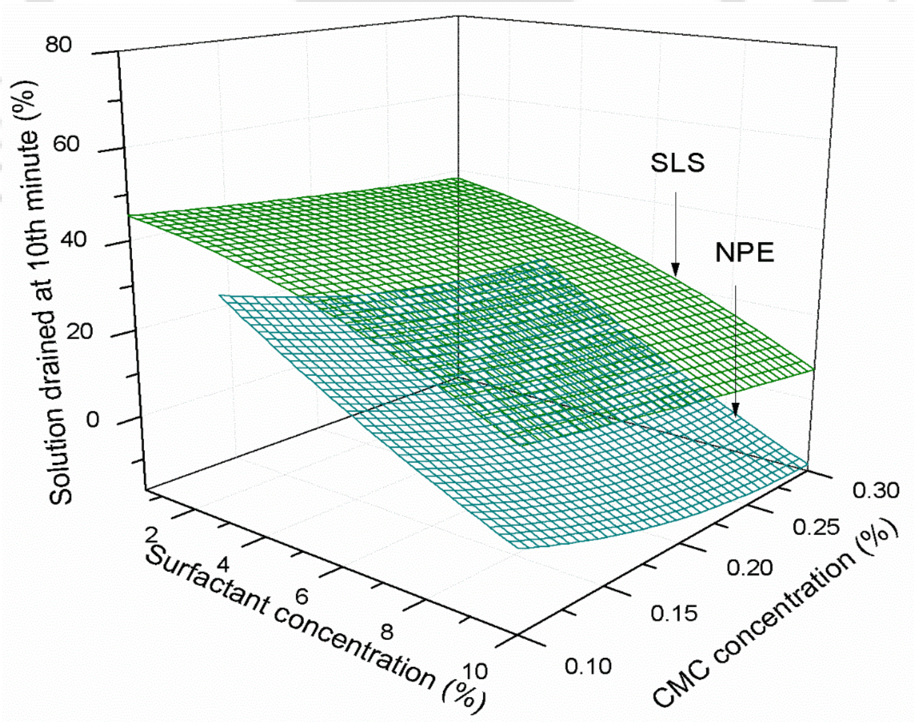
4.3.2 Response Surface Models for Foam Stability

On further examination of relative behaviour of foam stability, the percentage solution drained of NPE foam also reduces with increase in surfactant and CMC concentration, similar to earlier observations on SLS foam (Fig. 4.10). But the early drainage (5th minute) of NPE foam is very much lesser than that of SLS foam particularly when lower levels of surfactant and CMC concentration is adopted. For instance, foam produced with NPE surfactant shows 61% lesser drainage at 5th minute when compared to that of SLS at 3% and 0.1% surfactant and CMC concentration respectively. This can be attributed to the relatively higher viscosity of NPE and CMC surfactant solution when compared to that of SLS and CMC even at lower concentration levels. Furthermore, comparative studies on foam produced with and without CMC reveals the significant role of CMC on reduction in foam drainage (Fig. 4.11). In addition, the greater compatibility between NPE and CMC is also evident from the greater reduction in drainage of foam achieved due to addition of CMC to NPE surfactant solution. For instance, the addition of 0.3% CMC to 3% NPE surfactant solution results in 98% reduction of foam drainage when compared to surfactant solution without CMC. Another comparison on SLS and NPE foam without CMC indicates that, the drainage of SLS foam is relatively lesser than that of NPE foam. However on addition of CMC, the NPE foam preforms better than that of SLS foam in terms of stability again confirming the better compatibility between NPE and CMC.

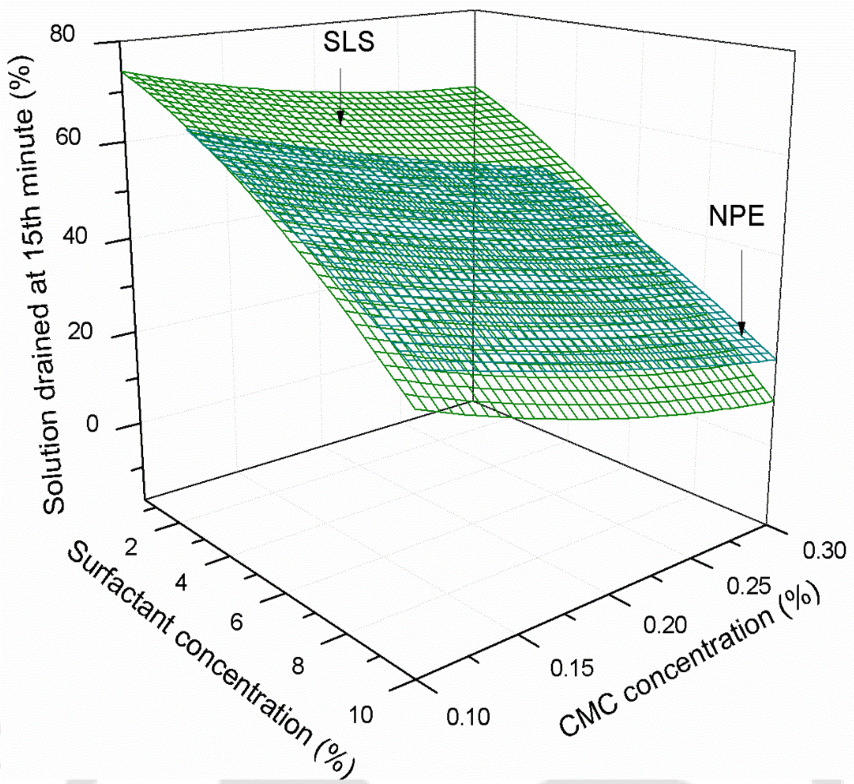
The implication of foam generation pressure on foam drainage is apparent in (Fig. 4.12) which shows that the drainage of NPE foam reduces significantly with increase in foam generation pressure similar to behaviour of SLS foam. Hence based on above observations, it is recommended to use higher surfactant and CMC concentration in addition to higher foam generation pressure in order to produce stable foam with lesser drainage.



(a) 5th minute



(b) 10th minute



(c) 15th minute

Fig. 4.10 Response surface plots for percentage solution drained for SLS and NPE (FGP 490 kPa)

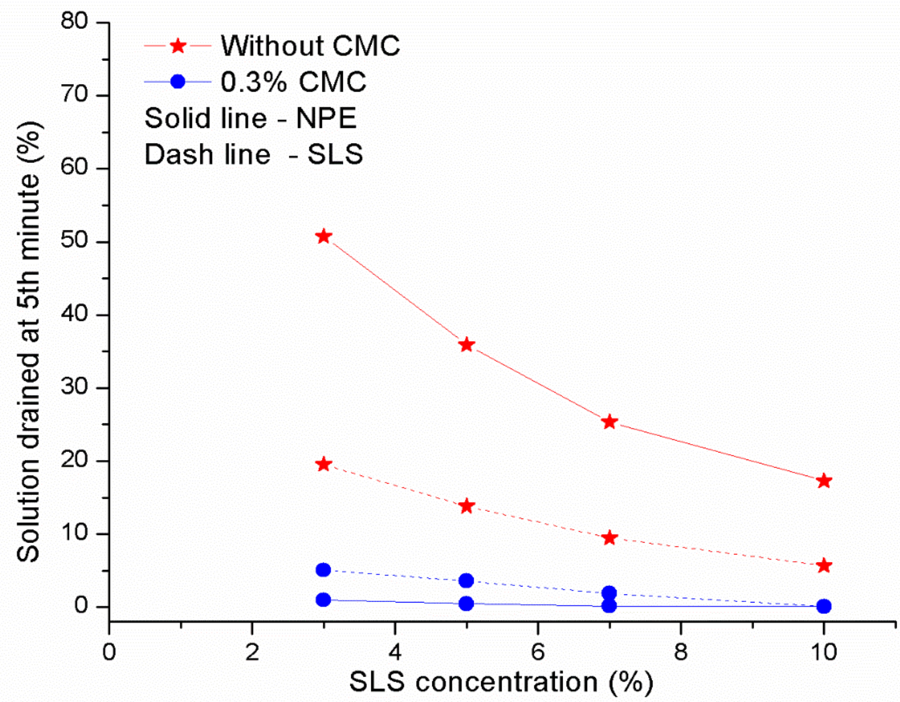


Fig. 4.11 Comparison of percentage solution drainage (at 5th minute) of foam produced with and without CMC for surfactants SLS and NPE

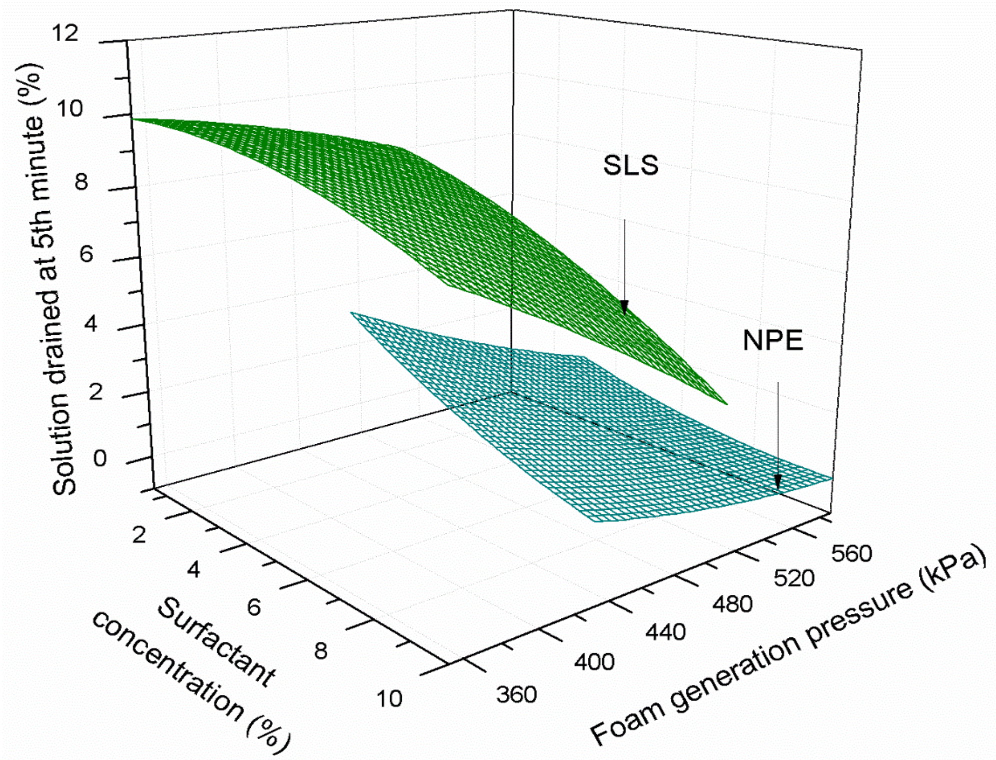


Fig. 4.12 Response surface plots for percentage solution drained (at 5th minute) for SLS and NPE (0.1% CMC)

4.3.3 Response Surface Model for Viscosity of Surfactant Solution

In line with earlier observations on SLS, the viscosity of NPE surfactant solution is also found to increase with increase in surfactant and CMC concentration (Fig. 4.13). However the increase in viscosity of NPE surfactant solution due to addition of CMC is very significant when compared to that of SLS. For instance, addition of 0.1% CMC to surfactant solution prepared with 10% NPE concentration results in 174% enhancement in viscosity when compared to that of SLS where addition of CMC results in 53% increase in viscosity (Fig. 4.14). The greater improvement in viscosity of NPE surfactant solution can be due to relatively more tight packing of surfactant molecules of NPE in presence of CMC. Moreover it is observed from literature, that generally non-ionic surfactants are more compatible with the additives due to the absence of charges. Also NPE being non-ionic, it is identified to be more compatible with additive CMC when compared to that of anionic surfactant SLS.

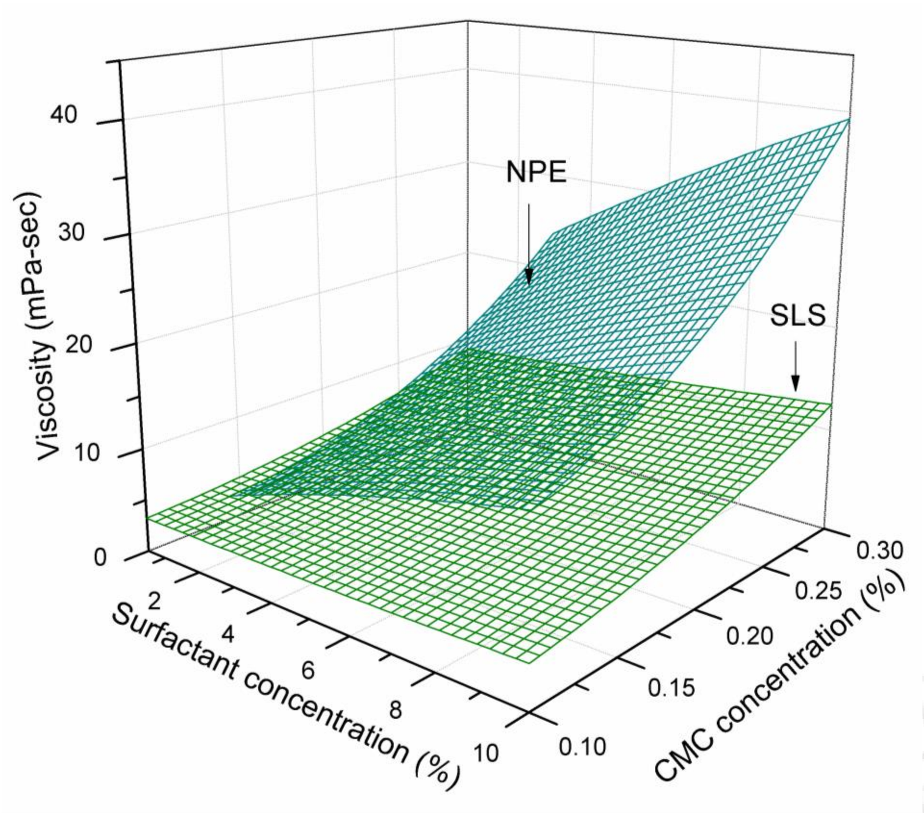


Fig. 4.13 Response surface plots for viscosity of surfactant solution for SLS and NPE

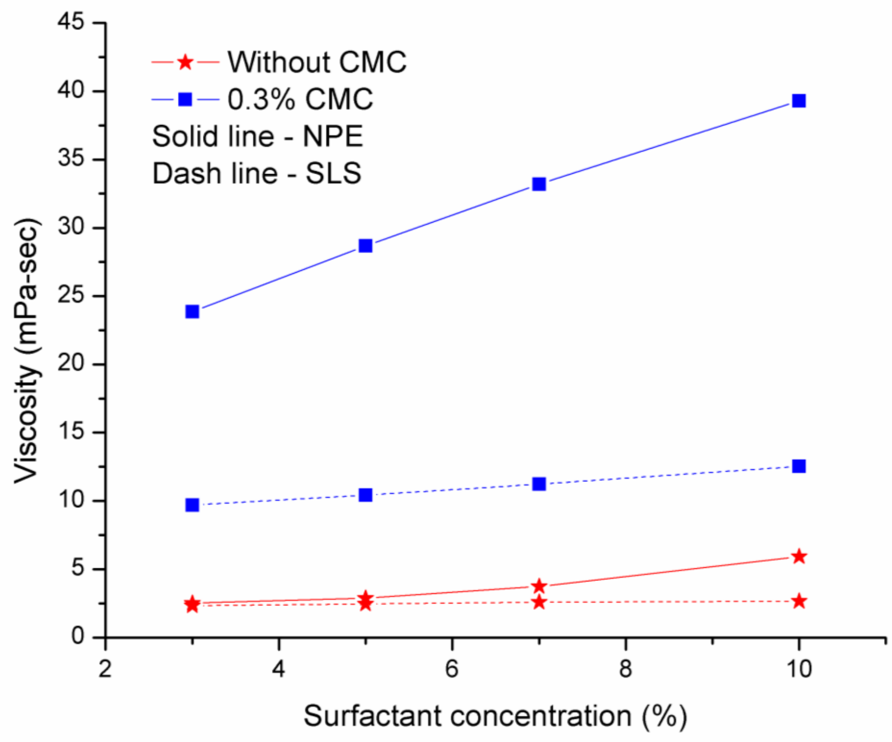


Fig. 4.14 Comparison of viscosity of surfactant solution prepared with and without CMC for surfactants SLS and NPE

4.3.4 Response Surface Models for Average Bubble Diameter of Foam

Fig. 4.15 shows the response surface plots for variation in average bubble size of foam produced with SLS and NPE surfactants. It is observed from Fig. 4.15 that the adoption of higher surfactant and CMC concentration results in foam with reduced bubble diameter for both SLS and NPE surfactants. For instance at least 40% reduction in average bubble diameter is observed when CMC is varied from 0 to 0.3% at 10% surfactant concentration (Fig. 4.16). The reduction in bubble size is attributed to the increase in viscosity of surfactant solution at higher levels of surfactant and CMC concentration as explained earlier. However the bubble sizes of both the SLS and NPE foams are found to be similar, though there is significant difference in their viscosities. For both the surfactants, the increase in FGP results in smaller size bubble microstructure (Fig. 4.17). Similar such observations were reported on influence of foam generation pressure on bubble size distribution of foam by Lee and Fan 2002; Nambiar 2006.

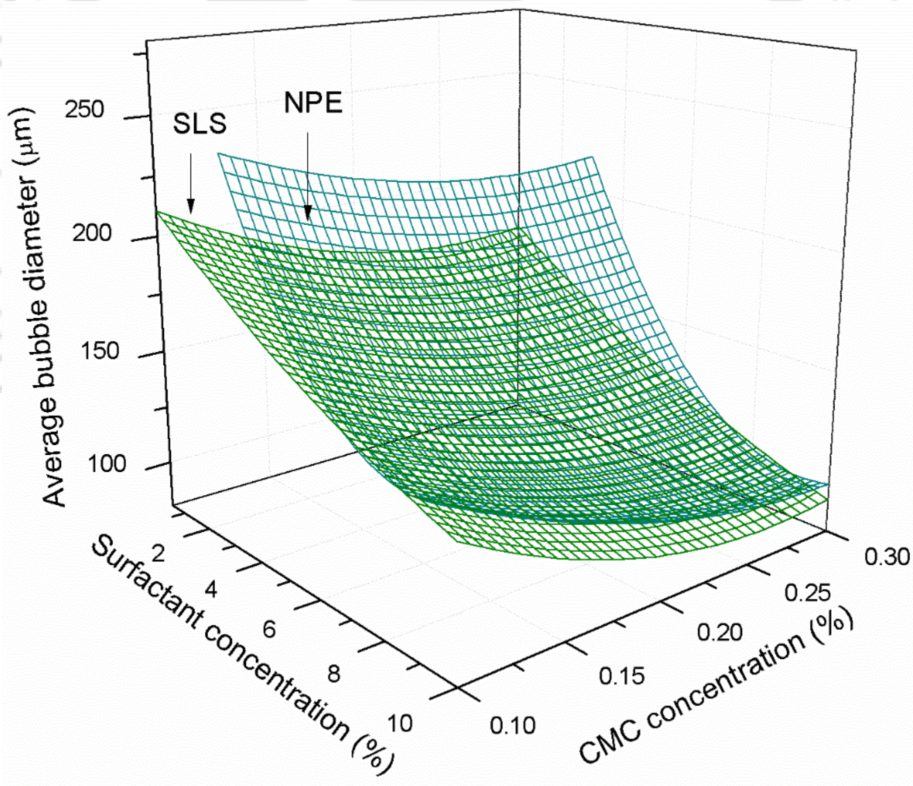


Fig. 4.15 Response surface plot for average bubble diameter for SLS and NPE (FGP 490 kPa)

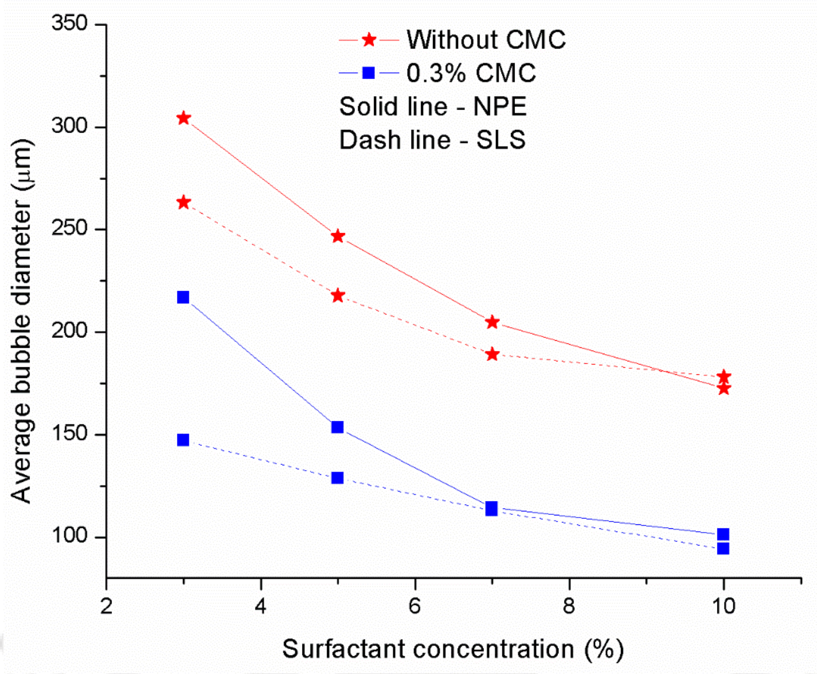


Fig. 4.16 Comparison of average bubble diameter of foam produced with and without CMC for surfactants SLS and NPE

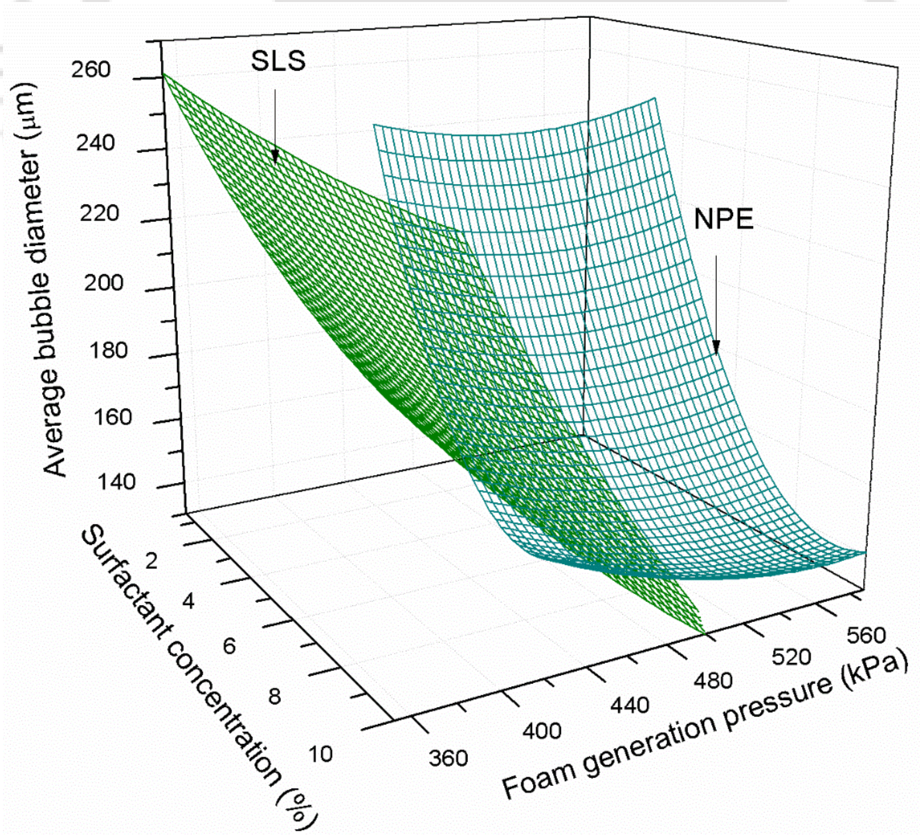


Fig. 4.17 Response surface plots for average bubble diameter for SLS and NPE (CMC 0.1%)

4.3.5 Relation between Initial Foam Density and Viscosity of Surfactant Solution

The effect of surfactant type on the relation between viscosity of surfactant solution and initial foam density is discussed below. It is observed that increase in surfactant concentration (for a constant CMC concentration) associated with some increase in viscosity results in reduction of the initial foam density for both the surfactants (Fig. 4.18). On the other hand significant increase in viscosity of surfactant solution achieved due to increase in CMC concentration (for a constant surfactant concentration) results in increase in initial foam density of NPE foam unlike SLS where reduction in foam density is observed. For instance a significant increase of 40% in initial foam density is observed due to increase in CMC concentration from 0.1 to 0.3% associated with 142% enhancement in viscosity of surfactant solution at a constant NPE concentration of 10%. Similar such increase in foam density was observed by Siva et al. (2015) when the viscosity of surfactant solution is increased due to increase in dosage of admixture. On contrary in SLS foam, 32% reduction in foam density is observed due to increase in CMC concentration (at constant SLS concentration of 10%). The reason for the difference in behaviour between the foam produced with these two surfactants can be attributed to the very high viscosity of NPE surfactant solution particularly at higher level of CMC concentration.

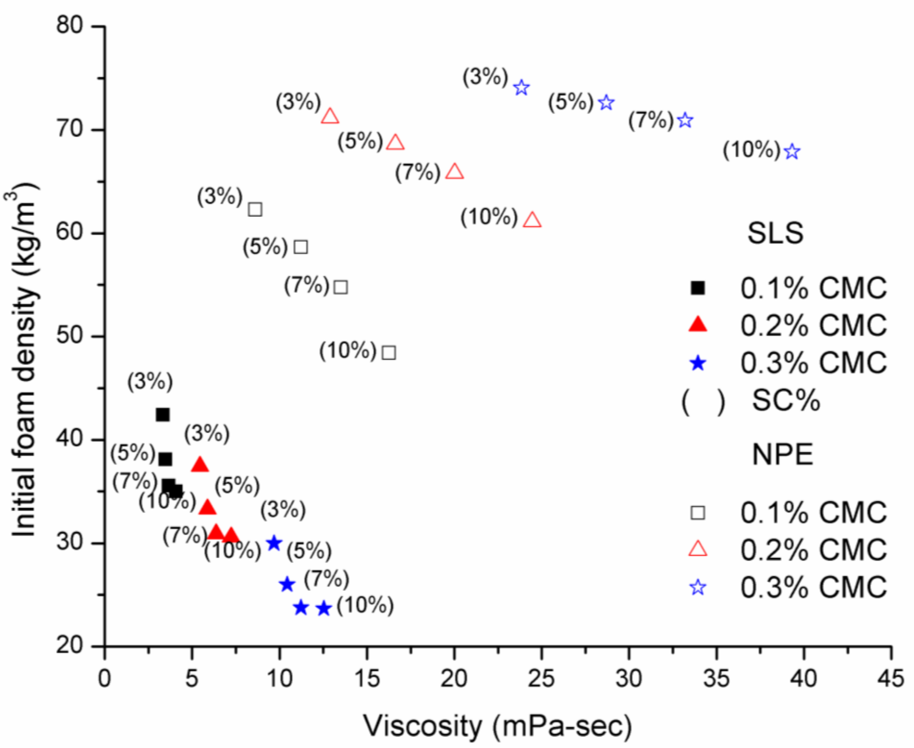


Fig. 4.18 Variation of initial foam density with viscosity of surfactant solution

4.3.6 Relation between Foam stability and viscosity of Surfactant Solution

A scatter plot showing the relation between viscosity of surfactant solution and percentage solution drained at 5th minute after foam generation for SLS and NPE surfactants are plotted in Fig. 4.19. As observed from Fig. 4.19, an increase in surfactant concentration (at a constant CMC concentration) associated with some increase in viscosity results in 81 to 98% reduction in percentage solution drained for both the surfactants. Furthermore the significant increase in viscosity of surfactant solution achieved due to increase in CMC concentration (for a constant surfactant concentration) results in greater reduction in foam drainage for both the surfactants with drainage less than 1% particularly at higher CMC concentration.

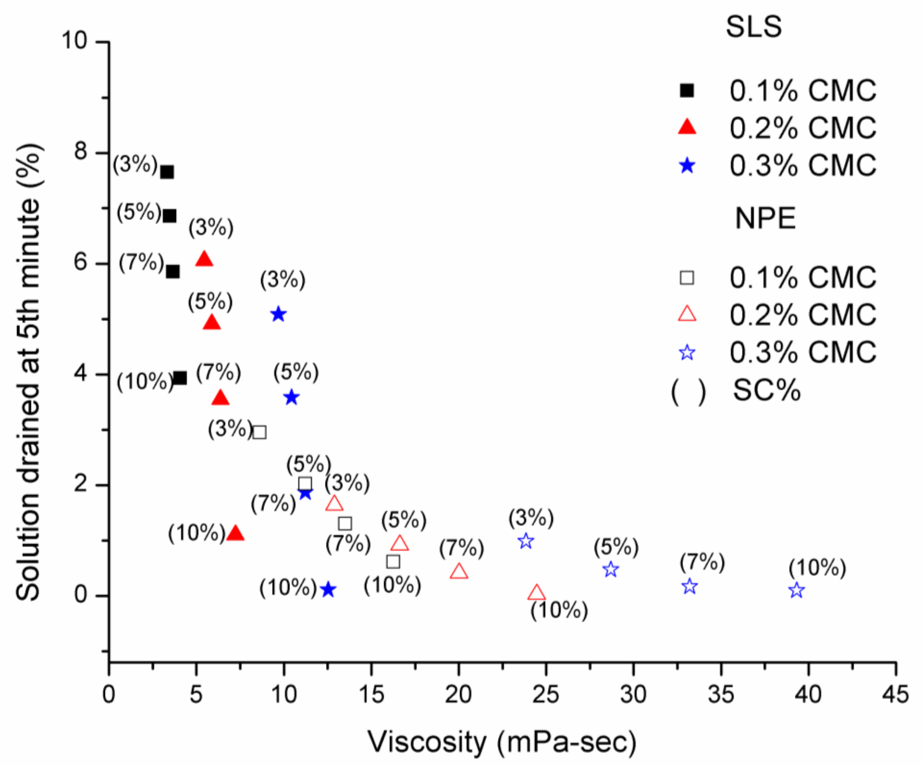


Fig. 4.19 Variation of solution drained with viscosity of surfactant solution for SLS and NPE

4.4 Optimization of Response Surface Models

Having studied the variation of foam characteristics with SLS, NPE, CMC concentration and foam generation pressure, the optimization of foam production parameters are carried out using Design Expert (version 10.0.6) as discussed below. Each response has been assigned an importance value (weightage) relative to the other responses. The criteria and the corresponding weightage assigned for optimization of both the surfactant and CMC concentrations are listed below.

(1) To achieve the initial foam density in the range of 32 to 64 kg/m^3 (as per ASTM C 796 requirement)
(2) To minimize foam drainage at 15th minute (maximum permissible drainage : 50%, weightage: 3/5)
(3) To minimize surfactant concentration (considering economy, weightage: 5/5)
(4) The maximum CMC concentration is limited to 0.2% (As the preliminary studies indicated that use of CMC concentration greater than 0.2% affects the stability and workability of foam concrete mixes)
(5) Foam generation pressure is fixed as 490 kPa (Adoption of pressure lesser than 490 kPa results in higher drainage, while beyond 490 kPa, it results in interrupted foam production)

Based on the optimization studies, the minimum NPE and CMC concentration at which the above mentioned criteria satisfied are identified as 6.1% and 0.16% respectively. The selection of above dosages are justified below. It is observed that at surfactant and CMC concentration lesser than 6% and 0.16%, though the foam meets ASTM specified initial foam density requirement, foam drainage at 15th minute is very high (greater than 50%). (Fig. 4.20 and Fig. 4.21). On the other hand adoption of surfactant concentration of 10%, though it results in foam with very less drainage, use of such high concentration is uneconomical for foam concrete production. Hence the choice of 6.1% NPE and 0.16% CMC is the economical dosage at which both initial foam density and drainage criteria are satisfied.

Similarly the optimum concentration of SLS and CMC are identified as 5% and 0.2% respectively (Fig. 4.20 and Fig. 4.21). Furthermore, the foam drainage of both the surfactants are found to be less than 5% in 300 seconds at the chosen optimum concentration levels. This drainage value is very low when compared to the value of 25% drainage obtained in time not less than 210 seconds as prescribed by Def Standard 42-40 (2002) for fire extinguishing foam.

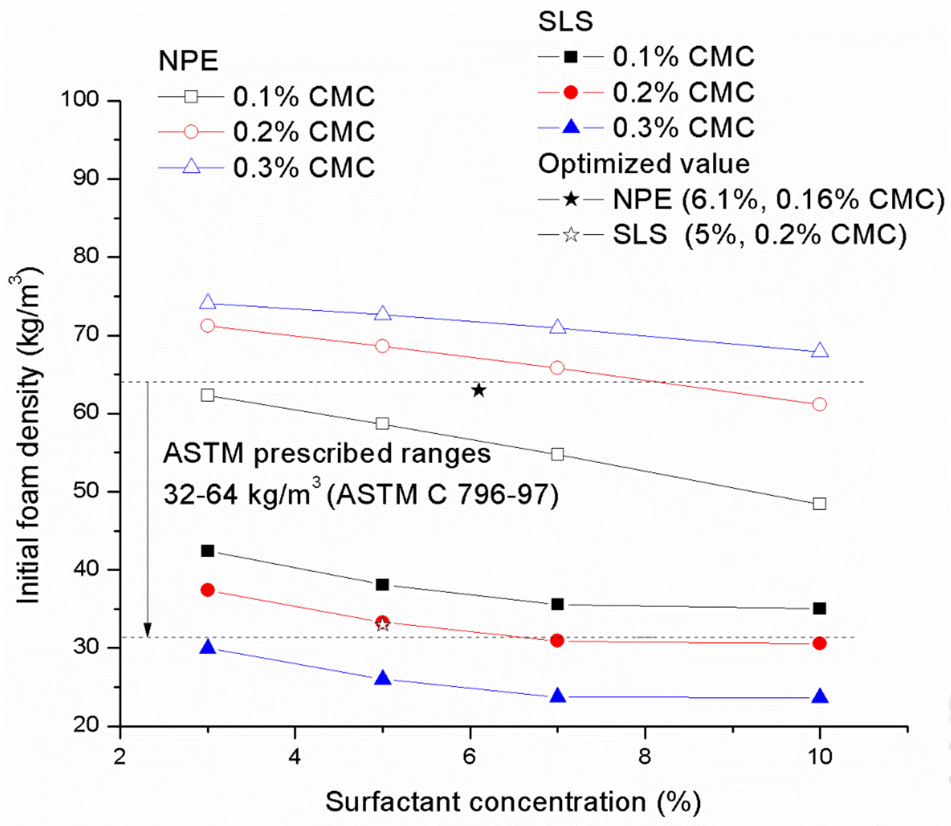


Fig. 4.20 Initial foam density of foam produced with surfactants SLS and NPE

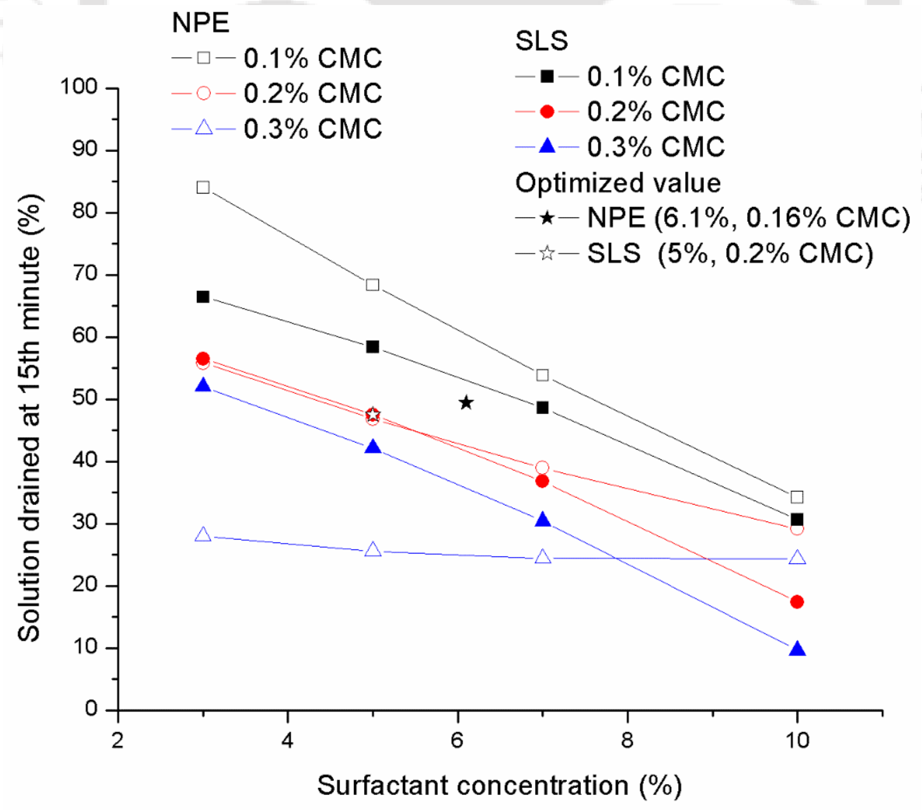


Fig. 4.21 Percentage solution drainage at 15th minute for surfactants SLS and NPE

At the optimum foam production parameters chosen, the initial foam density of NPE is very high when compared to SLS which can be attributed to the higher viscosity of NPE surfactant solution and greater compatibility between NPE and CMC (Table 4.7). However the bubble sizes are similar, though there is significant difference in viscosity between two surfactants.

Table 4.7 Optimized parameters and corresponding response goals

Foaming agent	Additive	SC (%)	CMC (%)	Foam generation pressure (kPa)	Initial foam density (kg/m^3)	Solution drained in 5^{th} minute (%)	Solution drained in 15^{th} minute (%)	Viscosity of surfactant solution (mPa-sec)	Average Bubble diameter (μm)
SLS	CMC	5	0.2	490	33	4.91	47.53	5.88	133
NPE	CMC	6.1	0.16	490	63	0.93	49.48	15.32	143

4.5 Stability of the Foam in the Mix

Having established the optimal surfactant concentration and foam generation pressure, as a next step, the suitability of these two surfactants and additive for the production of foam concrete need to be verified. Tests on foam cement paste of known design density 641 kg/m^3 and water-cement ratio of 0.58 need to carried out as per guidelines of ASTM C 796-97 to ascertain whether the requirements of ASTM C 869 with respect to fresh density, strength and water absorption of foam cement paste are fulfilled. ASTM C 796-97 has prescribed the following equations for arriving at the foam volume required for achieving a cement paste of known design density 641 kg/m^3 and water-cement ratio of 0.58.

$$V_f = 1000\ V_a / (1000 - W_{uf}) \text{ (per } m^3 \text{ of cement paste)} \quad (4.1)$$

$$V_a = (0.359^* W_{Tw} + 0.7965\ W_c) / 641\ m^3 \quad (4.2)$$

Where V_f = volume of foam; V_a = volume of air; W_{uf} = unit weight of foam; W_{Tw} = total weight of water; and W_c = weight of cement.

Foam concrete is made by mixing the cement slurry with a water-cement ratio 0.58 and preformed foam produced with the surfactants at the optimized SLS and CMC concentration of 5% and 0.2% respectively and optimized NPE and CMC concentration of 6.1% and 0.16% respectively and foam generation pressure of 490 kPa. The stability of test mixes is assessed by comparing the calculated and actual quantities of foam required to achieve a plastic density within ± 50 kg/m^3 of the design value and are summarized in Table 4.8 along with ASTM specifications. The foam concrete made with the foam produced with both the surfactants, at the optimized surfactant concentration and foam generation pressure, meets the physical requirements of ASTM, which confirms the foam stability. Also the actual quantity of foam required to attain the plastic density of 641 kg/m^3 within ±50 kg/m^3 of the design value is the same as the calculated quantity which again confirms the stability of the foam in the mix. Furthermore the performance of NPE in foam cement paste is relatively better in terms of compressive strength and water absorption which can be attributed to the improved foam characteristics of NPE in terms of higher foam density, lesser drainage and high viscosity of surfactant solution.

Table 4.8 Comparison of test results with ASTM specifications

Surfactant + additive	SC (%)	CMC (%)	FGP (kPa)	Initial foam density (kg/m^3)	Fresh density (kg/m^3)	Dry density (kg/m^3)	Compressive strength (MPa)	Water absorption (% by volume)	Actual/ calculated foam ratio
SLS + CMC	5	0.2	490	33	631	472	1.58	24	1
NPE + CMC	6.1	0.16	490	63	640	496	1.706	22.4	1
ASTM C 869-91 Requirements				32 to 64	641± 50	490± 40	1.40 (min)	25% (max)	1

4.6 Summary

Comparison of relative behaviour of foam produced using two different surfactants SLS and NPE is carried out through systematic evaluation of foam and surfactant properties such as initial foam density, foam stability, viscosity of surfactant solution and bubble size of foam through experiment design based on Response surface Methodology. The validity of response surface models developed for predicting the variation of foam characteristics as the function of foam production parameters are verified experimentally. Based on the comparative studies it can be concluded that NPE surfactant solution is highly viscous with higher initial foam density and relatively lesser early drainage when compared to that of

SLS. From the optimization studies, the optimal SLS and CMC concentrations are identified as 5% and 0.2% while the optimum NPE and CMC concentrations are 6.1% and 0.16% respectively. However for both the surfactants the optimum FGP is fixed as 490 kPa to get uninterrupted foam production.

The foam thus produced has been tested for suitability for making preformed foam concrete as per ASTM recommendations. The foam concrete made with the foam produced with both the surfactants, at the optimized surfactant concentration and foam generation pressure, meets the physical requirements of ASTM, confirming the foam stability. In addition, the performance of NPE in foam cement paste is relatively better in terms of compressive strength and water absorption which can be attributed to the improved foam characteristics of NPE in terms of higher foam density, lesser drainage and high viscosity of surfactant solution.

Chapter 5
RELATIVE WORKABILITY BEHAVIOR OF FOAM CONCRETE PRODUCED WITH DIFFERENT SURFACTANTS

5.1 GENERAL

The chapter 4 comprises of studies on relative performance of foam produced with two commercially available synthetic surfactants namely sodium lauryl sulphate (Anionic) and nonyl phenol ethoxylated (Non-ionic) with additive carboxymethyl cellulose (CMC). Having identified their potential as foaming agents, as a next step, the relative workability behaviour of foam concrete produced using these two surfactants and additive CMC is discussed in this chapter.

This chapter deals with the experimental investigations conducted to ascertain the influence of foam volume on the fresh state properties of foam concrete. The water-solids ratio required to produce stable and workable foam concrete of different design densities are determined. The influence of characteristics of surfactant on fresh concrete properties is also discussed.

5.2 MATERIALS AND METHODOLOGY

5.2.1 Materials

Foam is produced by aerating the two surfactants SLS and NPE with additive CMC using the foam generator. The optimum values of foam production parameters viz., SLS and CMC concentration, NPE and CMC concentration and foam generation pressure arrived at as 5 % and 0.2%, 6.1% and 0.16%, 490 kPa respectively through earlier optimization studies discussed in Chapter 4 have been adopted. Ordinary Portland cement of 43 grade conforming to (IS 8112) is used. For a given density, many researchers have reported that use of fine sand resulted in uniform microstructure of concrete leading to enhancement of strength (McCormick 1967; Nambiar and Ramamurthy 2006; Nambiar and Ramamurthy 2006a). Hence the river sand is pulverized and sand finer than 300µm (specific gravity 2.6) is used for the present study. A constant cement- sand ratio of 1:2 by weight is adopted throughout the study.

5.2.2 Mixture Composition and Mixing

Foam concrete mixes of three different design densities viz., 1000, 1500 and 1800 kg/m^3 are made by varying foam volume between 50% and 12%. The equation 5.1 has been prescribed by ASTM 796-97 to calculate the foam volume for a cement paste of design density 641 kg/m^3 and w/c ratio 0.58.

$$\text{Wet density, } 641\text{kg/m}^3 = (W_{Tw}+W_c) / [(W_{Tw}/1000)+((W_c/3.13*1000)+ V_a)] \quad (5.1)$$

Where W_{Tw} = weight of total water including the water present in the foam;

W_c = weight of cement

V_a = volume of air

After solving for the volume of air, the volume of foam (V_f) can be determined from the equation 5.2, arrived at by treating the diluted foam chemical as water;

$$V_f = V_a + (W_f / 1000) \quad (5.2)$$

$$\text{Where } W_f = W_{uf} * V_f \quad (5.3)$$

W_f = weight of foam

W_{uf} = unit weight of foam

The above equations for cement paste are appropriately modified by including the sand component (W_s is the weight of sand) and the foam volume required to achieve foam concrete design densities of 1000, 1500 and 1800 kg/m^3 are calculated as shown in the sample calculation procedure below for design density 1500 kg/m^3.

$$W_c + W_{Tw} + W_s = 1500 \quad (5.4)$$

$$W_c + 0.9W_c + 2W_c = 1500 \quad (5.5)$$

W_c = 385 kg/m^3

Having arrived at the cement content, the foam volume for mortar is determined in a way similar to that described for cement paste as follows;

Wet density, 1500 kg/m^3 = $(W_{Tw} + W_c + W_s) / [(W_{Tw}/1000) + ((W_c / 3.13*1000) + (W_s / 2.6*1000)) + V_a)]$ (5.6)

The details of mix proportions determined for various densities of foam concrete are presented in Table 5.1. The mixing sequence consist of first preparation of homogeneous base mix of cement and sand slurry in the mixer machine, followed by addition of calculated weight of foam to the base mix. The mixing is done till the foam is uniformly blended into the slurry. Based on cost analysis, it is observed that the cost of NPE surfactant and CMC at the optimized foam production parameters for one cubic meter of foam concrete production (design density 1500 kg/m^3), amounts to ₹ 1006. Similarly, the cost of SLS surfactant and CMC is found to be ₹ 425 for one cubic meter of foam concrete production. Hence SLS is found to be relatively economical when compared to that of NPE.

Table 5.1 Mix proportions for cement- sand foam concrete mixes

		Foam concrete			Mortar		
					Base mix -1	Base mix -2	Base mix -3
Target fresh density (kg/m^3)		1000	1500	1800	1921	1980	2044
Cement (kg/m^3)		246.9	384.6	480	482.3	513.2	546.3
Fine aggregate (pulverized sand) (kg/m^3)		493.8	769.2	960	964.7	1026.5	1092.7
Air volume equation (V_a)		$(0.681W_c+0.616W_s) / 1000$	$(0.521W_c+0.424W_s-0.5W_{Tw}) / 1500$	$(0.425W_c+0.308W_s-0.8W_{Tw}) / 1800$	NA	NA	NA
w/s		0.35	0.3	0.25	0.328	0.286	0.247
Foam volume V_f (%)	SLS (5%) + CMC (0.2%)	49	24	12	NA	NA	NA
	NPE (6.1%) + CMC (0.16%)	50	25	13			

W_c – weight of cement, W_s – weight of sand, W_{Tw} – weight of total water, V_a – volume of air, V_f – volume of foam

5.2.3 Test Procedure

The objective of the study is to identify the water solid ratio requirement to produce stable and workable foam concrete mixes. Stability of the mix is defined as the state of mix at which the density ratio (ratio of measured fresh density to design density) is one or nearly one without segregation or bleeding. The water-solid ratio required to produce stable foam concrete with actual fresh density within ±50 kg/m^3 of the desired design density is identified. As a next step the workability of the mixes need to be evaluated. As foam concrete is self-flowing in nature, workability test commonly conducted by slump test is not applicable and hence workability is assessed through through measurement of (i) spread in a flow table (ASTM C 230) and (ii) flow in modified Marsh cone (Jones et al. 2003).

5.3 Fresh State Properties of Foam Concrete

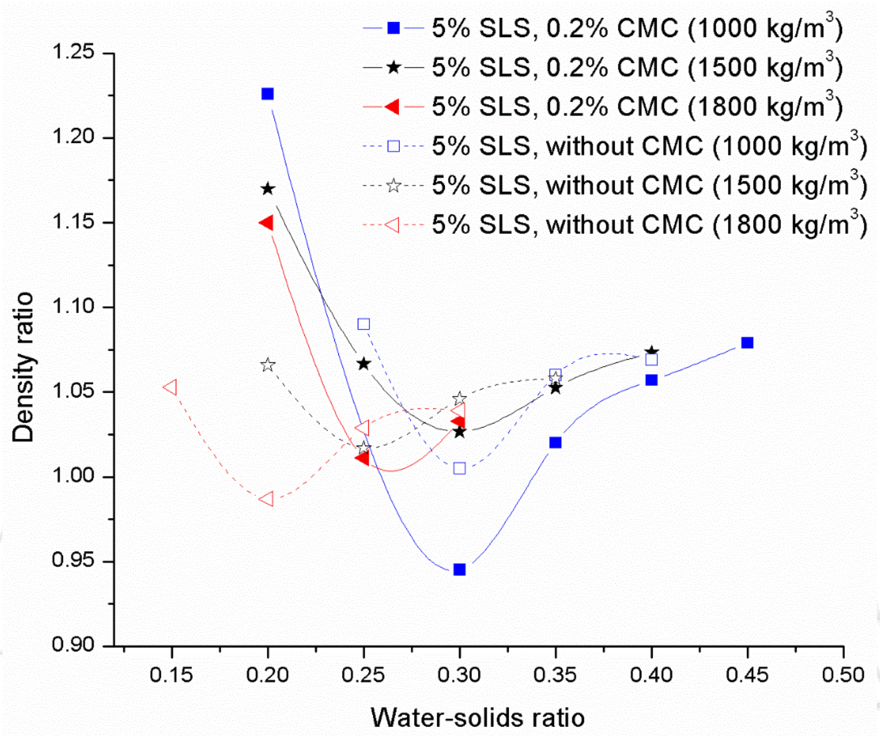
The important fresh state properties of foam concrete are flowability and self-compactability. It is reported that the foam concrete mix is classified as a stable mix if the achieved fresh density is within ±50 kg/m^3 of the desired design density. The water demand needed for stable concrete mix is determined by gradually increasing the water solids ratio to the point where design fresh density is achieved with no signs of segregation or bleeding. Hence in this case the water solids ratio is governed by the stability of the mix unlike the conventional (normal weight) concrete wherein the water solids ratio is decided based on strength. This section actually deals with the determination of optimum water- solids ratio to achieve stable foam concrete mixes of different design densities and studies on consistency of foam concrete mixes.

5.3.1 Determination of Optimum w/s ratio for Stable Concrete Mix

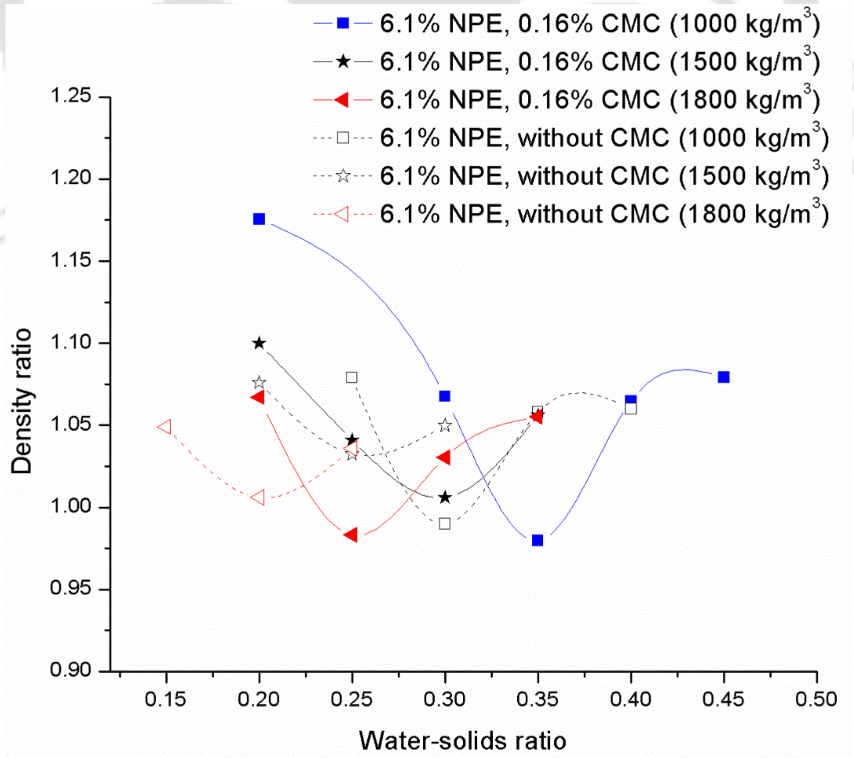
Fig. 5.1 shows the variation of density ratio with water-solids ratio for mixes with different design densities. It must be noted here that the water-solids ratio is very important to produce concrete mix of desired design density. This is because the use of lower water-solids ratios results in stiffer mixes causing breakage of bubbles. On the other hand use of higher water-solids ratio results in segregation of the foam from the mix thus causing instability of concrete mix.

The above observations are in agreement with findings of other researchers who had conducted similar studies (Nambiar and Ramamurthy 2008; SathyaNarayanan and

Ramamurthy 2012). Hence the optimum water-solids ratio within which measured density lies within ± 50 kg/m^3 of different design densities are determined as shown in Table 5.2.



(a) Sodium lauryl sulphate



(b) Nonyl phenol ethoxylated

Fig. 5.1 Variation of density ratio with water-solids ratio

Table 5.2 Water-solid ratio requirement for stable mixes for foam concrete produced with surfactants SLS and NPE

Design fresh density (kg/m^3)	Permitted range as per ASTM C 869-91	Identified water-solids ratio for stable mixes with CMC	Identified water-solids ratio for stable mixes without CMC
1000	1000 ± 50	0.35	0.3
1500	1500 ± 50	0.3	0.25
1800	1800 ± 50	0.25	0.2

Also the w-s ratio requirement is found to reduce marginally with increase in density indicating that the workability is influenced by the foam content. For mixes with higher foam volume the adhesion between bubbles and solid particles increases the stiffness of the mix resulting in higher water requirement to avoid breakage of bubbles during mixing.

Comparative studies on w/s ratio requirement for concrete mixes produced with and without CMC reveals that, both the surfactants (SLS and NPE) with CMC requires more water in order to achieve a particular target density within ± 50 kg/m^3. For instance, 17% increase in water demand is realized for production of stable concrete mix of design density 1500 kg/m^3, when CMC is added to surfactant solution. The reason for this behaviour can be attributed due to enhancement of viscosity of surfactant solution as a result of addition of CMC.

5.3.2 Consistency

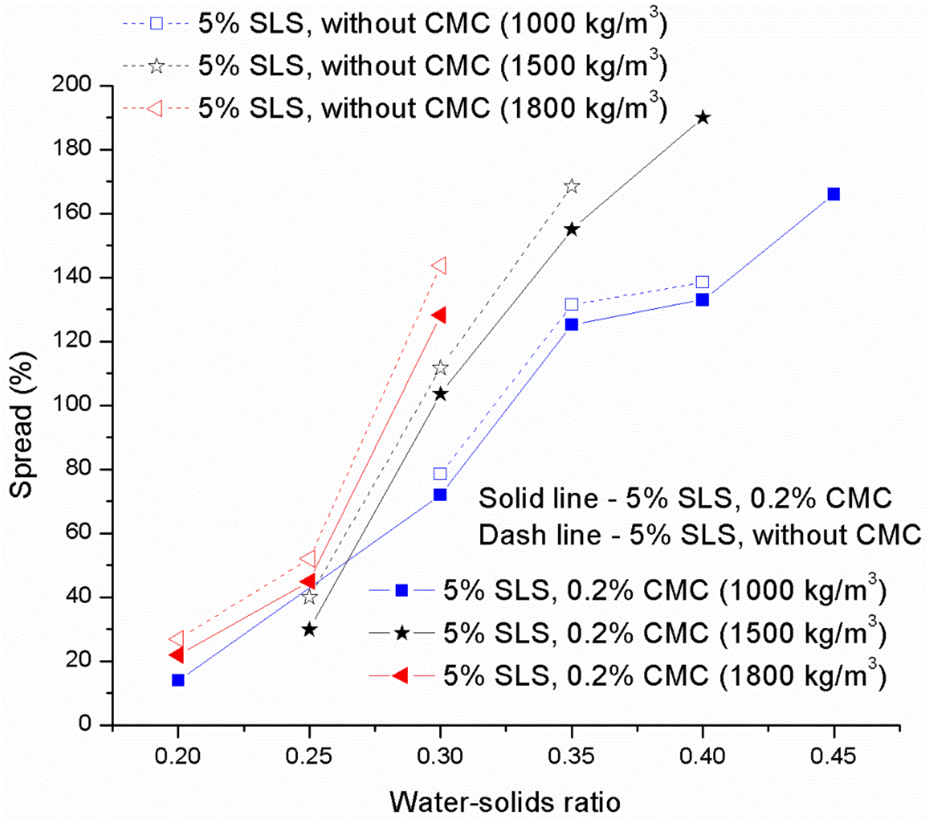
The consistency of the mix is very important to achieve the desired design density. Having identified the optimum water solids ratio required to produce stable mix, as a next step the workability of these mixes are to be studied. A spread flow of 45% was reported as an appropriate value of workability to confirm a good stability of foam concrete mix by (Nambiar and Ramamurthy 2008). To date, few research works have been undertaken to determine minimum workability required for desirable mix. Hence with the objective of determination of desired workability range for stable mixes, two workability measurement techniques viz., spreadability and marsh cone flow are adopted for measurement of workability.

5.3.3 Flow Spread Test

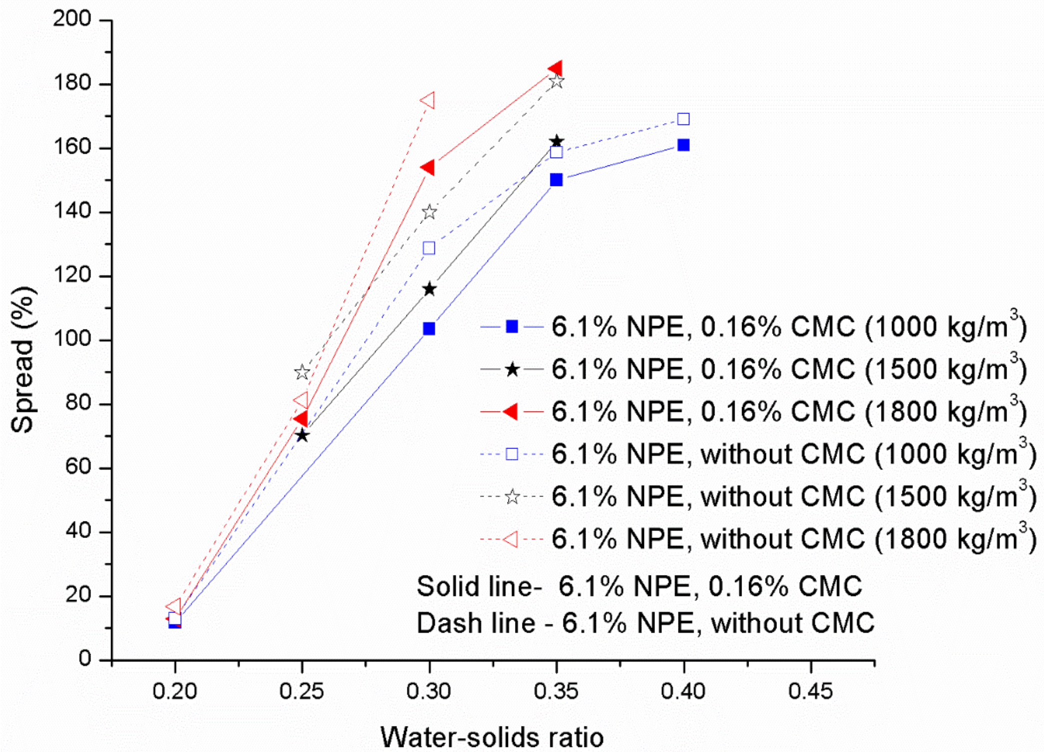
ASTM standard cone is used for the measurement of workability and the cone is filled with foam concrete soon after mixing, and is lifted and the average flow of concrete is measured without any shocking motion of the flow table as it would affect the entrained air bubbles. The spread of concrete is expressed as a percentage of the base diameter of the cone (Fig. 5.2).

Fig. 5.2 Flow cone test

For the foam concrete produced with both the surfactants, for a constant water-solids ratio, the spread reduces with an increase in foam volume (Fig. 5.3). Alternatively to achieve a particular workability, an increase in foam volume in the mix demands a higher water solid ratio. Hence, the foam volume has considerable influence on the flow behaviour of foam concrete. The addition of CMC which is basically a viscosity enhancing agent to surfactant solution results in significant enhancement in viscosity of surfactant solution as discussed in Chapter 4. The increase in viscosity of surfactant solution is also reflected in the rheological characteristics of foam concrete. Comparative studies on flow behaviour of foam concrete with and without CMC establishes that the addition of CMC results in 6% to 17% and 5% to 14% reduction in spread for surfactants NPE and SLS respectively. Hence the maximum dosage of CMC is fixed as 0.2% during optimization studies from the workability perspective. Based on the above studies it can be suggested that the optimum value of CMC need to be checked from point of view of workability and stability of foam concrete mixes also in addition to optimization studies on foam properties.



(a) Sodium lauryl sulphate



(b) Nonyl phenol ethoxylated

Fig. 5.3 Variation of Spread with water solid ratio

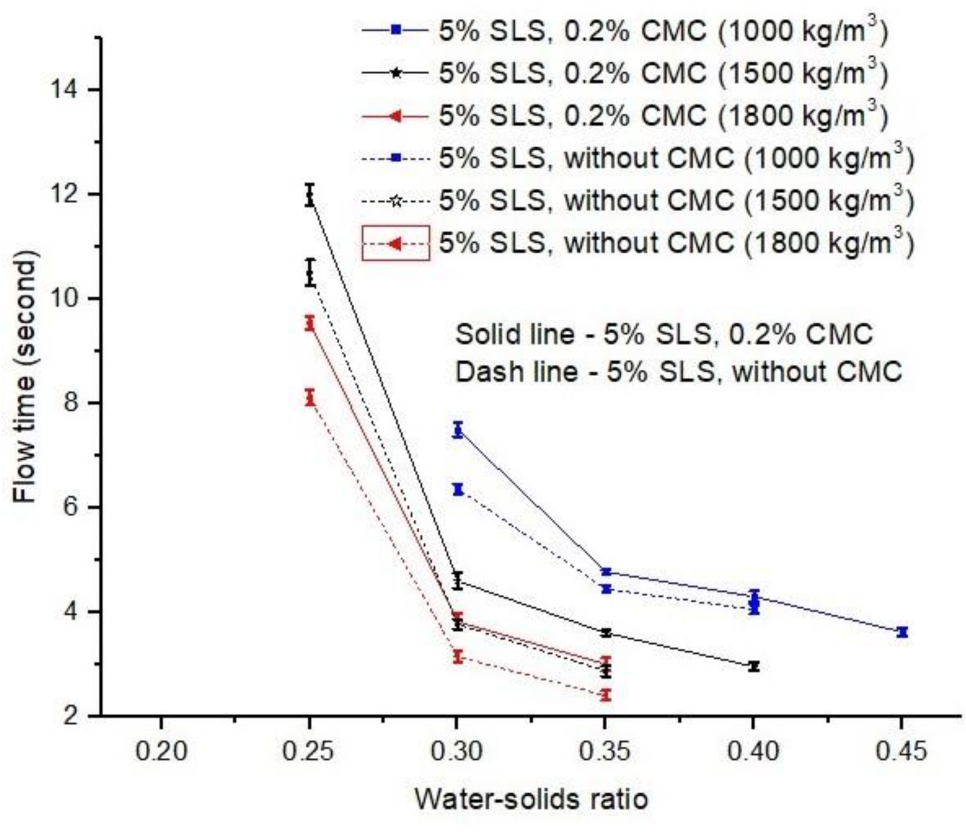
Comparative studies between two surfactants indicates that for constant water solid ratio the spread of foam concrete produced with NPE is 44% higher than that of concrete made with SLS. Although the viscosity of NPE surfactant solution is very high when compared to that of SLS, the flow behaviour of concrete shows a different picture. Hence the higher foam density of NPE can be attributed to the greater spread of foam concrete with NPE. The spreadability requirement to attain the desired design density ranges from 75 to 150% and 45 to 125% for surfactants NPE and SLS respectively.

Fig. 5.4 Modified Marsh cone test

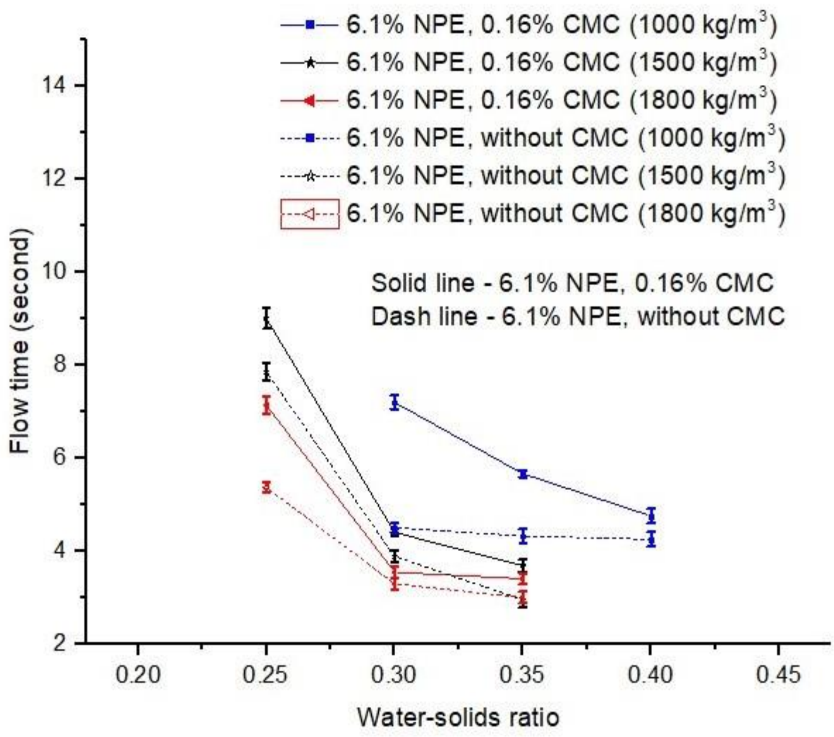
5.3.4 Marsh Cone Flow Test

The flowability, which depends on the fluidity of the mix, is determined using the marsh cone. It is a simple and practical method for obtaining a relative measure of the paste fluidity by measuring the time taken for a certain volume of paste to flow through a cone with a small opening (Fig. 5.4). This test is modified by Jones et al. (2003) in terms of orifice diameter (increased from 8 mm to 12.5 mm) and volume of efflux (1 litre instead of 200 ml) to take into account of the fine aggregate particles. The cone is filled with 1.5 litre of sample. The time taken for 1.0 litre of foam concrete to flow through the constricted orifice is measured. The marsh cone efflux time is an indication of the plastic viscosity, i.e., as the fluidity of the mix increases, the flow time decreases. Fig. 5.5 shows the variation of flowability of stable foam concrete mixes with w/s ratio for different design densities of concrete. For a constant water-solids ratio, the flow time increases with an increase in foam volume. In line with earlier observations an increase in flow time of 8 to 60% and 18 to 22% is observed for surfactants NPE and SLS respectively due to addition of CMC which again proves that the addition of CMC affects the workability related characteristics of foam concrete. The flow time requirement to produce stable mix varies from 4.4 to 7.1 seconds and 4.6 to 9.5 seconds for NPE and SLS respectively.

As expected, for the range of design plastic densities considered, greater spread values corresponds to shorter flow times (hence reduced plastic viscosity), thereby indicating a relationship between these two properties of foam concrete (Fig. 5.6).



(a) Sodium lauryl sulphate



(b) Nonyl phenol Ethoxylated

Fig. 5.5 Variation of flow time with water solid ratio

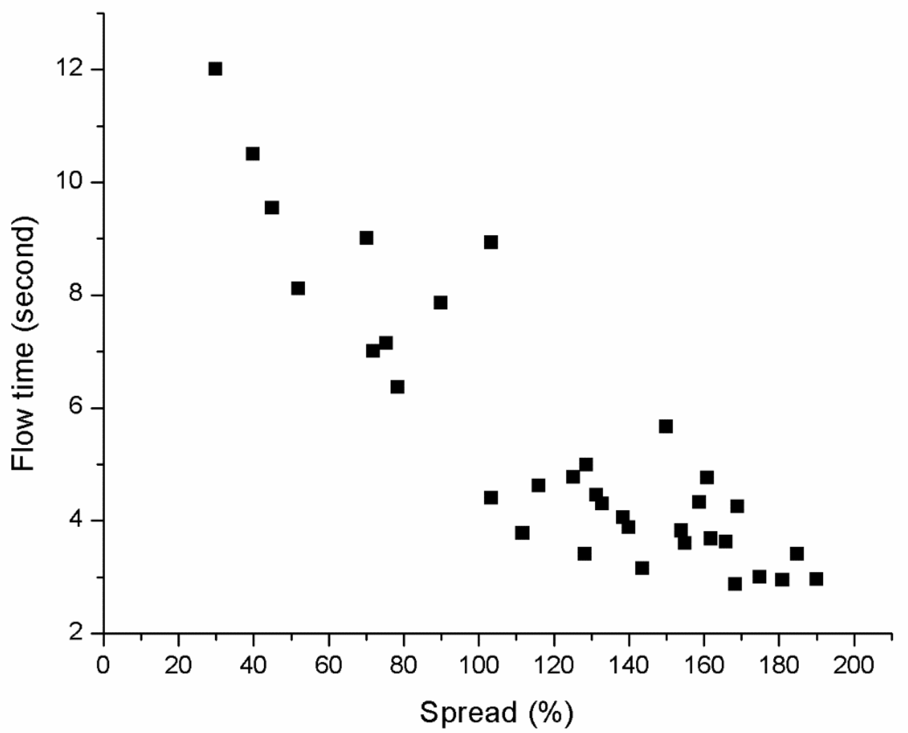


Fig. 5.6 Variation of flow time with spread

5.3.5 Effect of Surfactant Concentration on Workability of Foam Concrete

The role of surfactant concentration on foam related characteristics have been explained in detail in chapter 3 and 4. Since the surfactant concentration affects the viscosity of surfactant solution, hence it can also affect the workability related characteristics of foam concrete. Hence studies on the influence of surfactant concentration on spreadability and flowability of foam concrete mix of particular design fresh density 1500 kg/m^3 is carried out as discussed below. Firstly the surfactant concentration is varied from 3 to 10% at optimum CMC concentration of 0.2% and 0.16% for surfactants SLS and NPE respectively. Also in order to eliminate the effect of w/s ratio on workability, the w/s ratio is fixed at 0.3. Studies indicates that increase in surfactant concentration results in decrease in spread and increase in flow time of foam concrete mixes (Fig. 5.7 and Fig. 5.8). For instance, increase in surfactant concentration from 3% to 10% results in 10% and 7% reduction in spread for foam concrete made with surfactants NPE and SLS respectively. Further, the flow time of concrete mixes increases by 46% and 48% for foam concrete produced with surfactants NPE and SLS respectively due to increase in surfactant concentration. The reason for reduction in spread and increase in flow time can be attributed to the significant increase in viscosity of surfactant solution and considerable reduction in liquid fraction of foam due to increase in surfactant concentration.

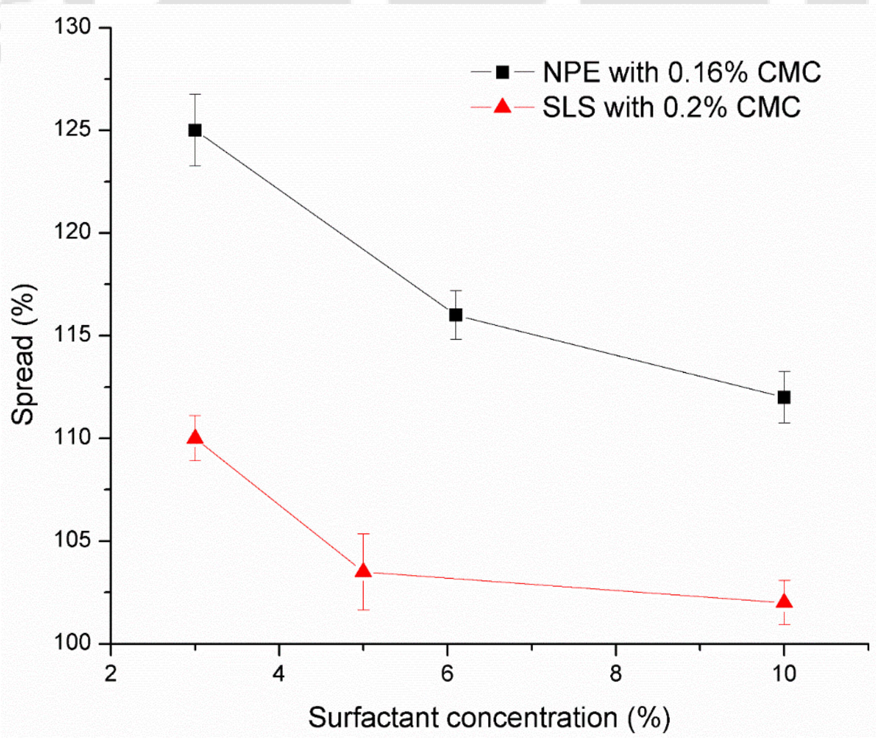


Fig. 5.7 Variation of Spread with surfactant concentration

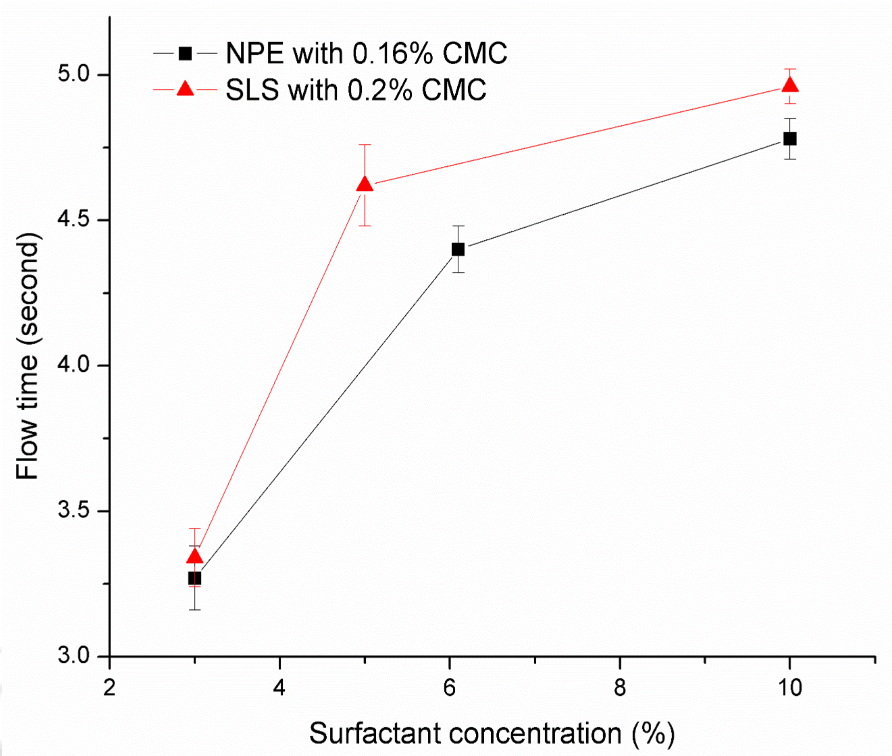


Fig. 5.8 Variation of flow time with surfactant concentration

As a next step the influence of variation of CMC concentration on workability of concrete is studied at optimum surfactant concentration of 5% and 6.1% for SLS and NPE respectively. Studies indicates that increase in CMC concentration results in gradual decrease in spread and increase in flow time of foam concrete mixes. For instance, increase in CMC concentration from 0.1 to 0.3% results in 17% and 14% reduction in spread for foam concrete made with surfactants NPE and SLS respectively (Fig. 5.9). Further, the flow time of concrete mixes increases by 49% and 43% for foam concrete produced with surfactants NPE and SLS respectively due to increase in CMC concentration (Fig. 5.10). The reason for reduction in spread and increase in flow time can be attributed to the significant increase in viscosity of surfactant solution and reduction in liquid fraction of foam due to increase in CMC concentration.

Hence, the optimization of dosages of surfactant and additive should also consider the stability and workability related characteristics of concrete in addition to foam characteristics.

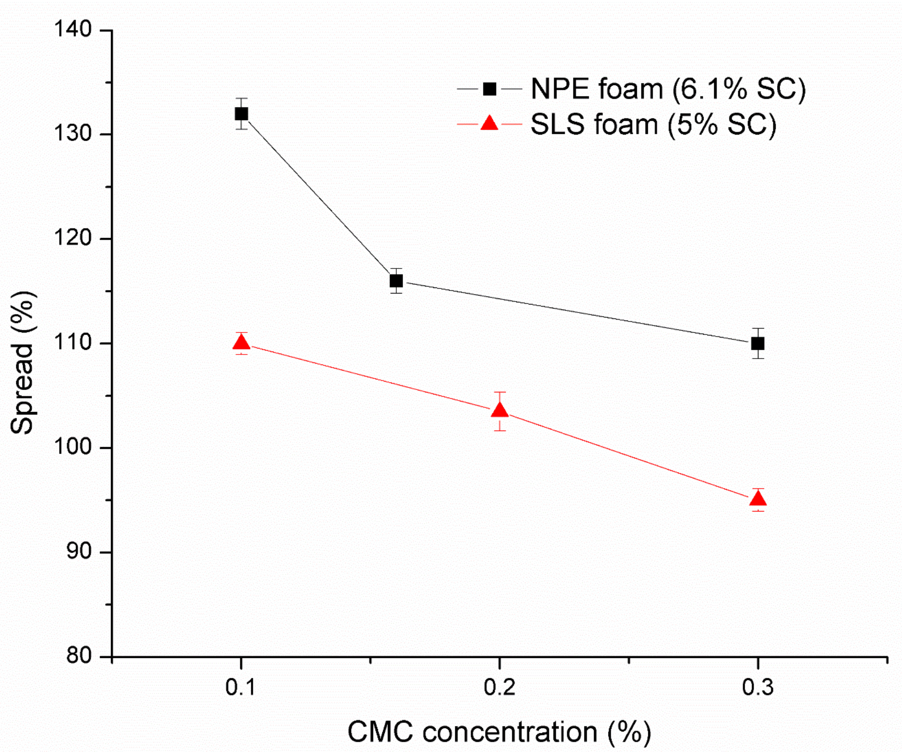


Fig. 5.9 Variation of Spread with CMC concentration

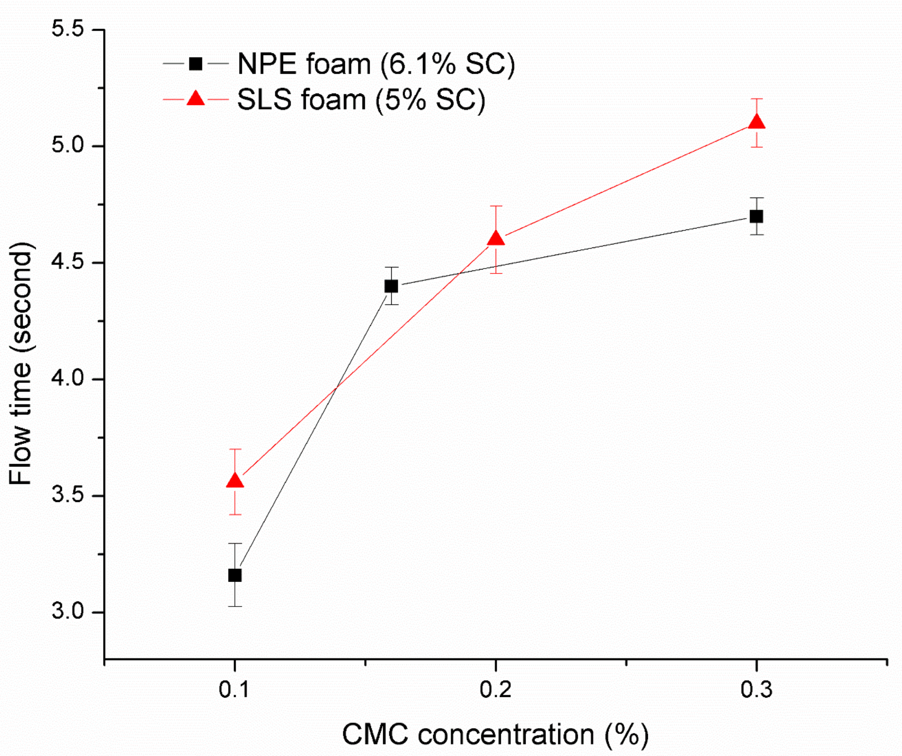


Fig. 5.10 Variation of flow time with CMC concentration

5.4 SUMMARY

The relative workability of foam concrete produced with two synthetic surfactants SLS and NPE have been studied. The water-solids ratio which would satisfy both the stability and consistence requirement of the mix for different design densities has been determined for concrete with two different surfactants. Furthermore the effect of addition of CMC on workability and water-solids ratio requirement is also studied. For the foam concrete produced with both the surfactants, for a constant water-solids ratio, the spread reduces and flow time increases with an increase in foam volume. Also the w-s ratio requirement is found to reduce marginally with increase in density indicating that the workability is influenced by the foam content. Experimental studies have established that the increase in surfactant and CMC concentration which increases the viscosity of surfactant solution also affects the workability related characteristics. Hence the optimum value of surfactant and CMC concentration need to be checked from point of view of workability and stability of foam concrete mixes also in addition to optimization studies on foam properties. The spreadability requirement to attain the desired design density ranges from 75 to 150% and 45 to 125% for surfactants NPE and SLS respectively. Similarly the flow time requirement to produce stable mix varies from 4.4 to 7.1 seconds and 4.6 to 9.5 seconds for NPE and SLS respectively. For a constant water solid ratio the spread of foam concrete made with NPE is 44% higher and flow time is 7% lower than concrete made with SLS.

Chapter 6
AIR VOID PARAMETERS AND ITS INFLUENCE ON COMPRESSIVE STRENGTH AND MOISTURE MOVEMENT CHARACTERISTICS OF FOAM CONCRETE

6.1 GENERAL

Having studied the effect of variation of foam production parameters on foam characteristics, and its effect on workability of concrete, as a next step its impact on the microstructure of foam concrete, compressive strength and water permeation characteristics need to be studied. The pore system of foam concrete consists of water filled voids (gel and capillary pores) and artificially entrained or entrapped air voids of size greater than 50 micrometres. Earlier studies have proved that the large capillary pores (of the order of 50 to 10000 nm) and extra-large pores (>10000 nm) are only influential on strength and permeability characteristics, whereas small pores (< 10 nm) and medium capillary pores (10 to 50 nm) are influential on drying shrinkage and creep of foam concrete (Jitchaiyaphum et al. 2013). The influence of shape and orientation of the pores on the mechanical properties of aerated concrete have been studied by (Cabrillac et al. 2006; Cabrillac and Malou 2000; Nambiar and Ramamurthy 2007). Further it has been reported earlier by researchers that foam concrete mixes with narrower air void size distribution showed higher compressive strength (Kearsley and Visagie 2002; Nambiar and Ramamurthy 2007). Narayanan and Ramamurthy (2000) investigated the paste–void interface of aerated concrete in relation to paste aggregate interface in normal concrete and observed that larger pores in aerated concrete can be treated as aggregates of zero density. Few researchers have made attempts to characterize the spacing of air-voids in air-entrained concrete by developing spacing equations (Attiogbe 1993; Philleo 1983). Based on the review of literature it is observed that the studies available on the influence of air void characteristics on the mechanical properties and durability related characteristics of foam concrete is very limited. Hence the aim of this investigation is to study the influence of type of surfactant, addition of additive, foam characteristics, and density of concrete on the microstructure of concrete. Further the correlation of air void characteristics to compressive strength and water permeation characteristics are also investigated.

6.2 AIR VOID CHARACTERIZATION

6.2.1 Selection of Air void Characterization Method

ASTM C 457 has prescribed few methods such as Linear traverse method, modified point count method and paste air ratio modification method for air void characterization of concrete. In linear traverse method, the parameters of air void system are determined by measurement of total length traversed and length traversed through air voids in concrete sample with series of regularly spaced lines in one or more planes intersecting the sample. In modified point count method, the procedure consists of observation of the frequency with which areas of a given component coincide with a regular grid system of points at which stops are made to enable the determination of composition. Air void system is determined based on total number of stops and the number of stops in the air voids. In some instances where sample is not representative of whole concrete, then paste air ratio method gives sensible results than methods mentioned above. However, it is to be noted that the above methods are more time consuming (ASTM C 457).

Adding to above there are also other direct and indirect measurement techniques available for the characterization of pore structure of cement-based material. Aligizaki (2005) have reviewed the various measurement techniques which included indirect methods such as mercury porosimetry, gas adsorption, and thermoporometry, and direct method like microscopy coupled with computer based image processing techniques. In indirect methods an external stimulus is applied to the material and the pore parameters are evaluated indirectly from measured responses such as adsorption, pressure for mercury intrusion etc. For instance in Mercury intrusion porosimetry (MIP) method, the amount of pressure required for mercury to intrude into the pores is inversely related to the size of pores. MIP generates volume and pore size distributions from the pressure versus intrusion data generated by the instrument using the Washburn equation. In Brunauer Emmett Teller (BET) surface area method, the specific surface area is determined from the volume of gas needed to form a monolayer on the surface of the sample. The major limitation of MIP and

BET techniques is that interpretation of results involves approximate assumption about pore shape and also the equipment is expensive (Lange et al. 1994). Furthermore, the foam concrete being weaker, specimens cannot withstand the high pressure employed in the above mentioned methods. Hence optical microscopy with image analysis method is adopted for the current research, since the method is proved to be both simple and accurate. Furthermore, it has been reported in literature that direct observation of pore structure by capturing images through optical and electron microscopes provide the pore shape directly (Lange 1994). Computer based image processing has been reported to be successfully used for pore characterization of cement based materials (Chatterji and Gudmundsson 1977; Kearsley and Visagie 2002; Lange et al. 1994; Petrov and Schlegel 1994; Pleau et al. 2001). This chapter discusses the investigations made to characterize the void system of foam concrete of three different design densities viz., 1000, 1500 and 1800 kg/m^3 produced with two synthetic surfactants by processing and analyzing the images of the prepared surfaces of specimens captured by an optical microscope.

6.2.2 Specimen and Surface Preparation

The materials and their properties and mix proportioning have been described in the earlier chapter under section 5.2.1 and 5.2.2. Foam concrete mixes of three different fresh densities viz., 1000, 1500 and 1800 kg/m^3 are made by varying foam volume between 50% and 12%. For each density, 4 cube specimens of size 50 mm are cast and moist-cured for 28 days. The cubes are then cut into slices of size 50 x 50 x 25 mm using a diamond rotary by cutting slices parallel and perpendicular to the cast surface and at different depths of specimen. Such slicing yields 6 specimens (cut surfaces) of size 50 x 50 x 25 mm, for each mix, as shown in Fig. 6.1.

It is to be noted for the successful application of image analysis, the quality of the surface treatment of specimen is very important for production of accurate results. However the conventional surface preparation method of filling pores with synthetic resin and smoothening by grinding is not suitable for foam concrete because resin impregnation can affect the physical structure of concrete (Petrov and Schlegel 1994). Hence the specimen surface preparation method prescribed by ASTM C 457 for contrast enhancement method is followed in the present study. For the preparation of specimens after slicing, the cut surface of each specimen is carefully lapped by abrasive paper of fineness grade 1000 using a polishing machine to obtain a plane surface on which the boundaries of the air-voids and matrix are sharp and easily distinguishable.

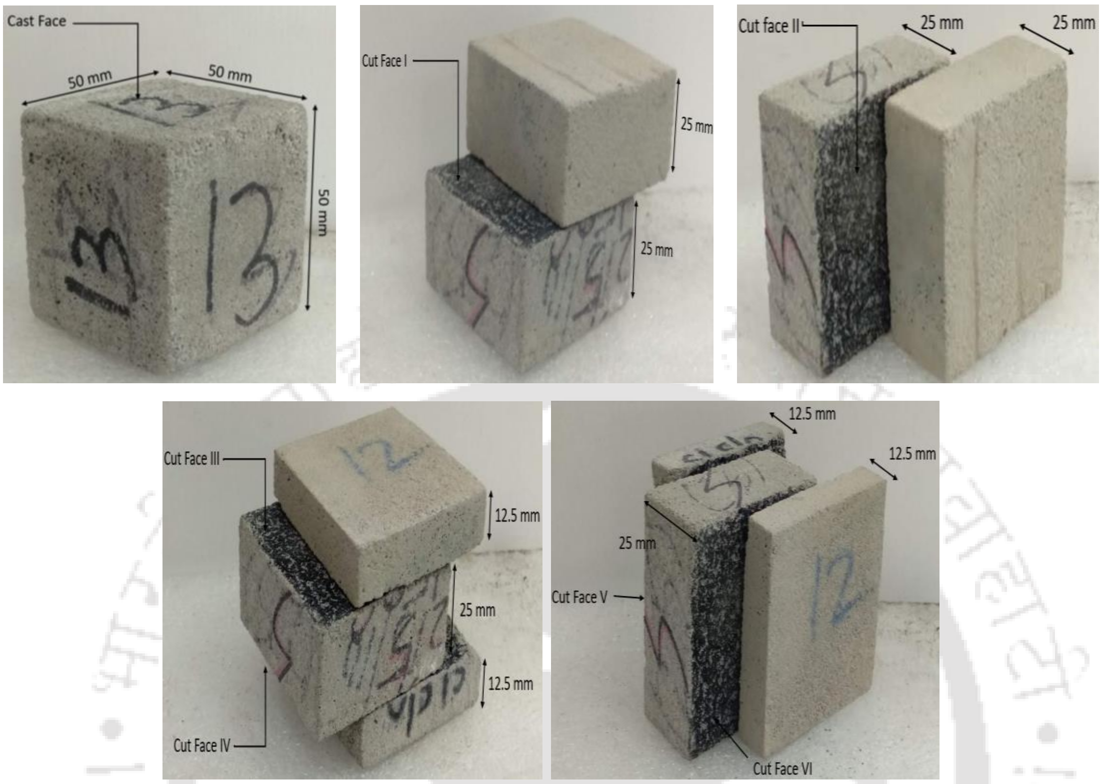


Fig. 6.1 Preparation of specimens for image analysis

Then using compressed air, cleaning of specimens are carried out and further dried in the oven at 50^{o}C for 3 days to ensure a dry surface for microscopic analysis. After this in order to further prepare the specimen to enable clear distinction between voids and remaining area, two coats of black ink are applied on the surface using permanent marker pen and was allowed to dry. After the ink had dried, the white powder is spread over the surface and worked into the air voids by means of fingertip. Then most of the excess powder is wiped away with the edge of razor blade and with a lightly oiled fingertip. Hence the specimen is now ready for measurement with voids appearing white and remaining area is black in colour and can be placed on the specimen support of an optical microscope for capturing the image.

6.2.3 Image Analysis Test Setup

The macro pores of foam concrete which are considered as voids with diameter greater than 50 µm are analysed using an optical microscope and a computer with image analysis software (Image J, 2006). A magnification of 0.7 X is selected with a pixel representing

0.162 microns which is determined through calibration. A total of 36 images are captured altogether at random locations from the six prepared surfaces for each foam concrete mix. Each image is digitized and converted into binary form as shown in Fig. 6.2 and Fig. 6.3.

6.3 Determination of Characteristics of Air void System

After the above processes, the main air void characteristics such as air void size distribution and shape of pores in terms of circularity were computed using the image analysis software. The assumption that all the air voids are spherical will not hold good for mixes with higher foam volume where irregular voids are observed due to merging of bubbles. Hence the feret diameter which is the longest distance between two parallel tangential lines drawn to air void was determined to define the air void size (Image J 2006).

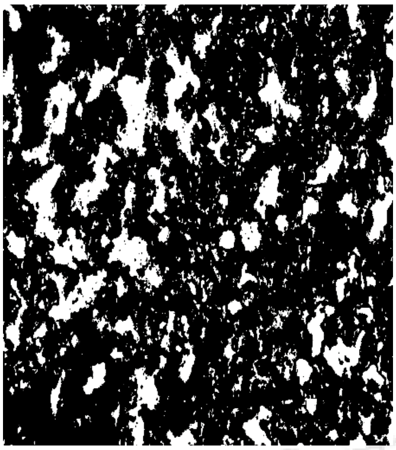
(i) With 0.16% CMC (ii) Without CMC

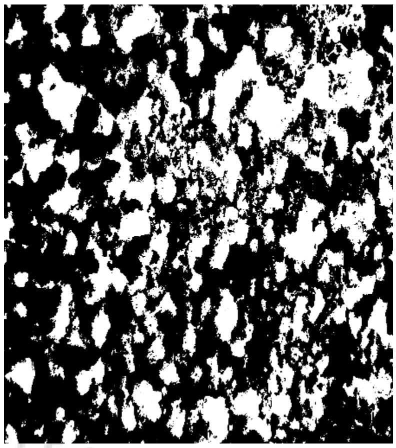
(a) Foam concrete with fresh density 1000 kg/m^3

(i) With 0.16% CMC (ii) Without CMC

(b) Foam concrete with fresh density 1800 kg/m^3

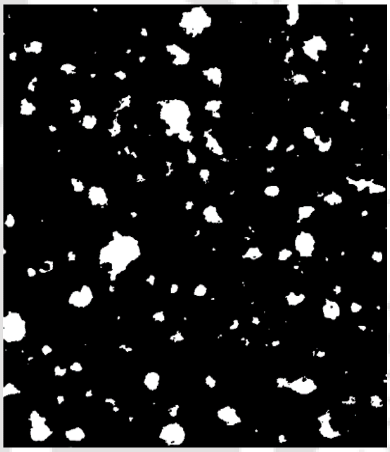
Fig. 6.2 Typical binary images of foam concrete produced with NPE (6.1%)





(i) with 0.2% CMC (ii) without CMC

(a) Foam concrete with fresh density 1000 kg/m^3



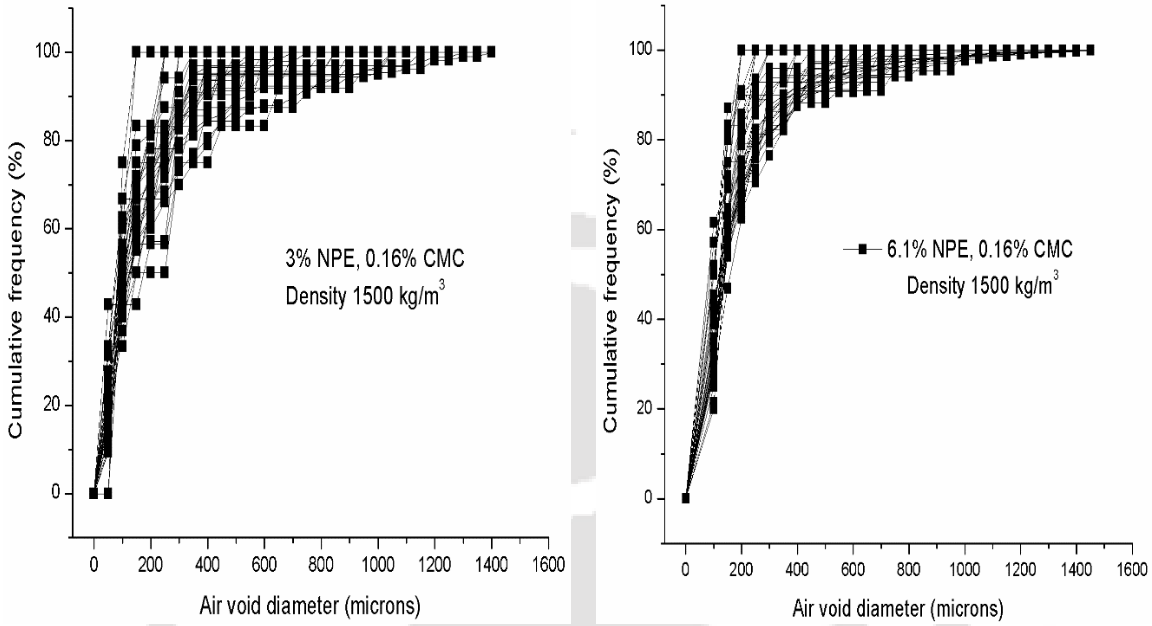
(i) with 0.2% CMC (ii) without CMC

(b) Foam concrete with fresh density 1800 kg/m^3

Fig. 6.3 Typical binary images of foam concrete produced with SLS (5%)

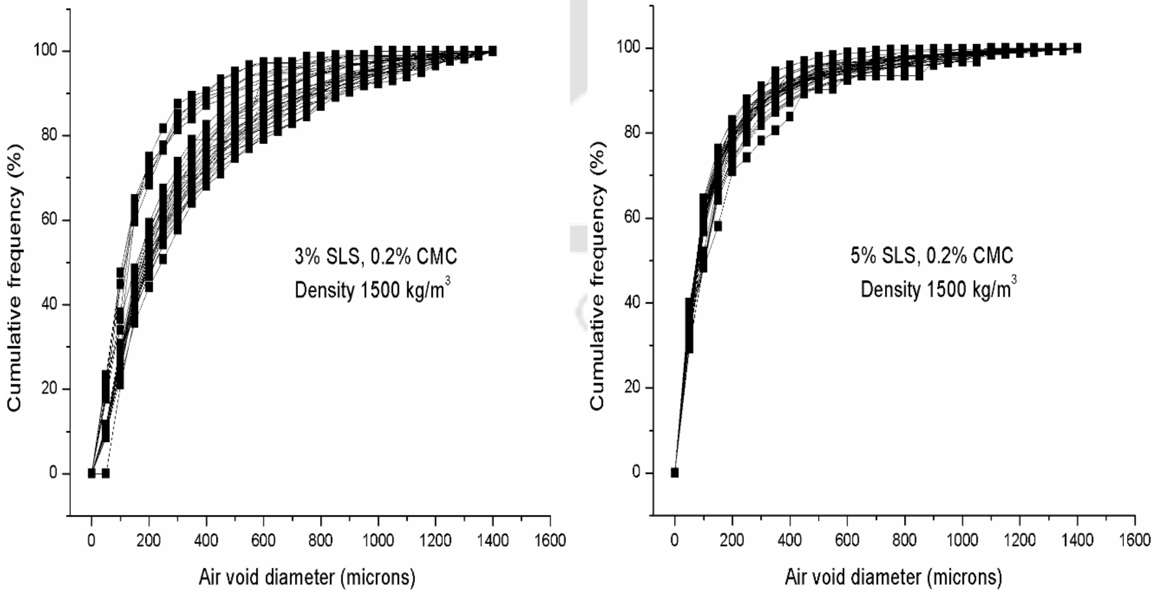
6.3.1 Air-void Size Distribution in Foam Concrete

Firstly the influence of the role of surfactant concentration on air void size distribution in concrete need to be studied. The air void size distribution in hardened concrete is determined by measurement of Feret diameter and presented in Fig. 6.4 and Fig. 6.5. Fig. 6.4 (a) and Fig. 6.4 (b) shows the air void size distribution in foam concrete produced with 3% NPE, and 6.1% NPE concentration respectively. Similarly Fig. 6.5 (a) and Fig. 6.5 (b) presents the air void size distribution in foam concrete produced with 3% SLS and 5% SLS concentration respectively. The histogram plots representing the air void size distribution for both the surfactants are shown in Fig. 6.6 and Fig. 6.7. In order to quantify and compare the air void size distribution of different mixes, the parameters used are D50 (median air void) and D90 (10% oversize air void) which can be read from the Fig. 6.4 and Fig. 6.5. Table 6.1 shows the comparison of air void size distribution for foam concrete produced with two different concentrations of surfactants SLS and NPE with additive CMC. Results indicates that increase in surfactant concentration results in significant reduction in air void size for both the surfactants (Table 6.1).



(a) 3% NPE, 0.16% CMC (b) 6.1% NPE, 0.16% CMC

Fig. 6.4 Variation of air void size distribution in foam concrete with surfactant concentration (NPE)



(a) 3% SLS, 0.2% CMC (b) 5% SLS, 0.2% CMC

Fig. 6.5 Variation of air void size distribution in foam concrete with surfactant concentration (SLS)

For instance, D90, the size of larger air voids, ranges from 406 to 917 microns in foam concrete produced with 3% SLS and 0.2% CMC concentration compared to size range of 310 to 495 microns for foam concrete produced with 5% SLS and 0.2% CMC concentration. The values 698 and 334 microns presented in Table 6.1 represents the overall arithmetic mean of D90 values derived for 36 images for foamed concrete with 3% and 5% SLS concentrations with 0.2% CMC respectively. Similar to above observations, a reduction of 24% and 44% in D50 is observed for foam concrete produced with NPE and SLS respectively when surfactant concentration is varied from lower to higher level as seen in Table 6.1.

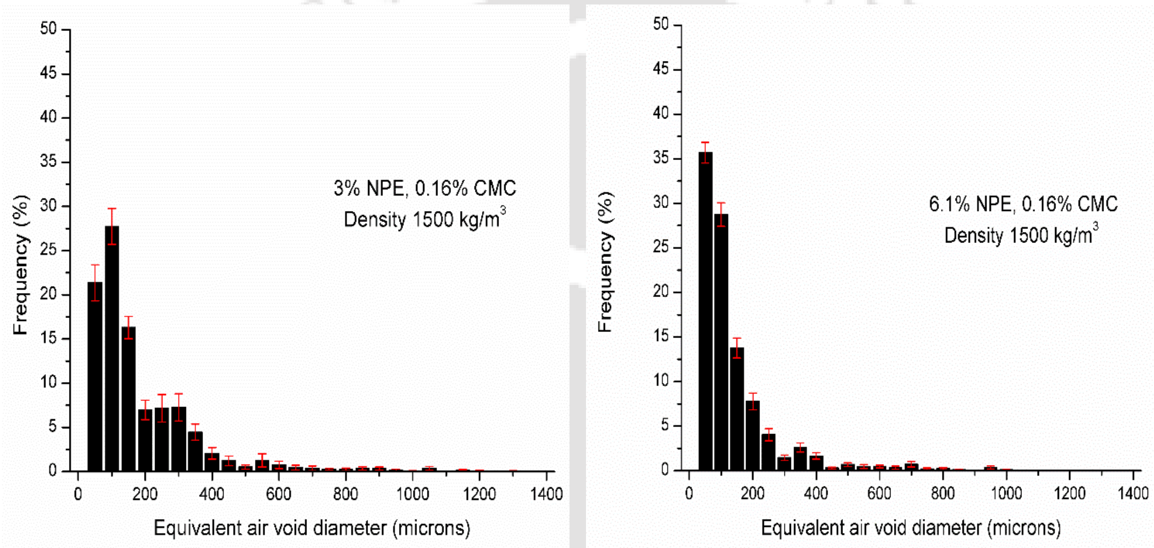


Fig. 6.6 Histogram plots showing variation in air void size distribution with surfactant concentration (NPE)

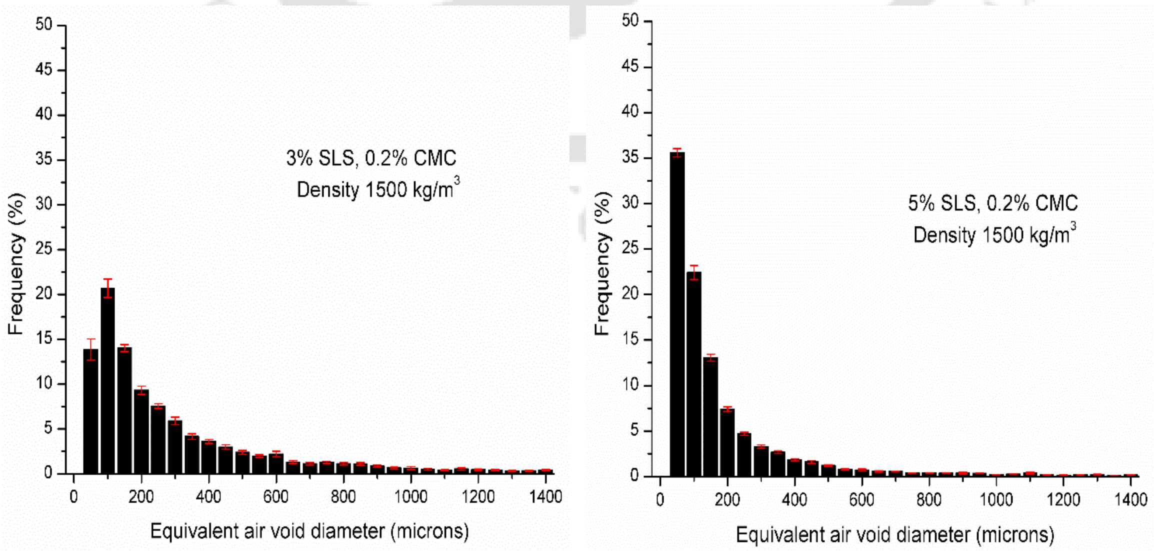


Fig. 6.7 Histogram plots showing variation in air void size distribution with surfactant concentration (SLS)

A similar trend is observed during analysis of bubble microstructure of foam also as discussed earlier in chapter 3 and 4. The above reduction in air void size at higher surfactant concentration can be attributed to significant increase in viscosity of surfactant solution at higher surfactant concentration for surfactants SLS and NPE.

Comparative studies on two surfactants indicates that the effect of lower surfactant concentration on microstructure is more apparent in SLS surfactant with very high values of D50 and D90. Further for both the surfactant concentrations studied, the air void size distribution is relatively smaller for foam concrete with NPE when compared to that of SLS. Particularly at 3% surfactant concentration D50 and D90 values of foam concrete with NPE are lower than that of SLS by 29% and 45% respectively. The reduction in air void size of foam concrete with NPE can be again attributed to the higher viscosity of NPE surfactant solution when compared to that of SLS. Hence it is observed that adoption of higher surfactant concentration is needed for a good microstructure with smaller air void size distribution in concrete.

Table 6.1 Effect of surfactant concentration on air void size distribution of foam concrete (fresh density 1500 kg/m^3)

Parameters	3% NPE, 0.16% CMC	6.1% NPE, 0.16% CMC	3% SLS, 0.2% CMC	5% SLS, 0.2% CMC
D50	142µ (100-200 µ)	108 µ (81-133 µ)	199µ (111-288 µ)	111 µ (98-149 µ)
D90	386µ (160-780 µ)	299 µ (150-510 µ)	698µ (406-917 µ)	334 µ (310-495 µ)

As a next step, the variation of microstructure of concrete with density of concrete (foam volume) and the influence of addition of CMC on air void size distribution is investigated as presented below. The frequency distributions of air void sizes and histogram plots are plotted for concrete produced with both the surfactants for different densities ranging from 1000 to 1800 kg/m^3 (Fig. 6.8 to Fig. 6.11). Table 6.2 and Table 6.3 shows the air void size distribution for foam concrete with three different design densities, for two surfactants (NPE and SLS) at optimized surfactant concentrations. Also comparison of air void sizes for foam concrete mixes with and without additive CMC is presented in Table 6.2 and Table 6.3.

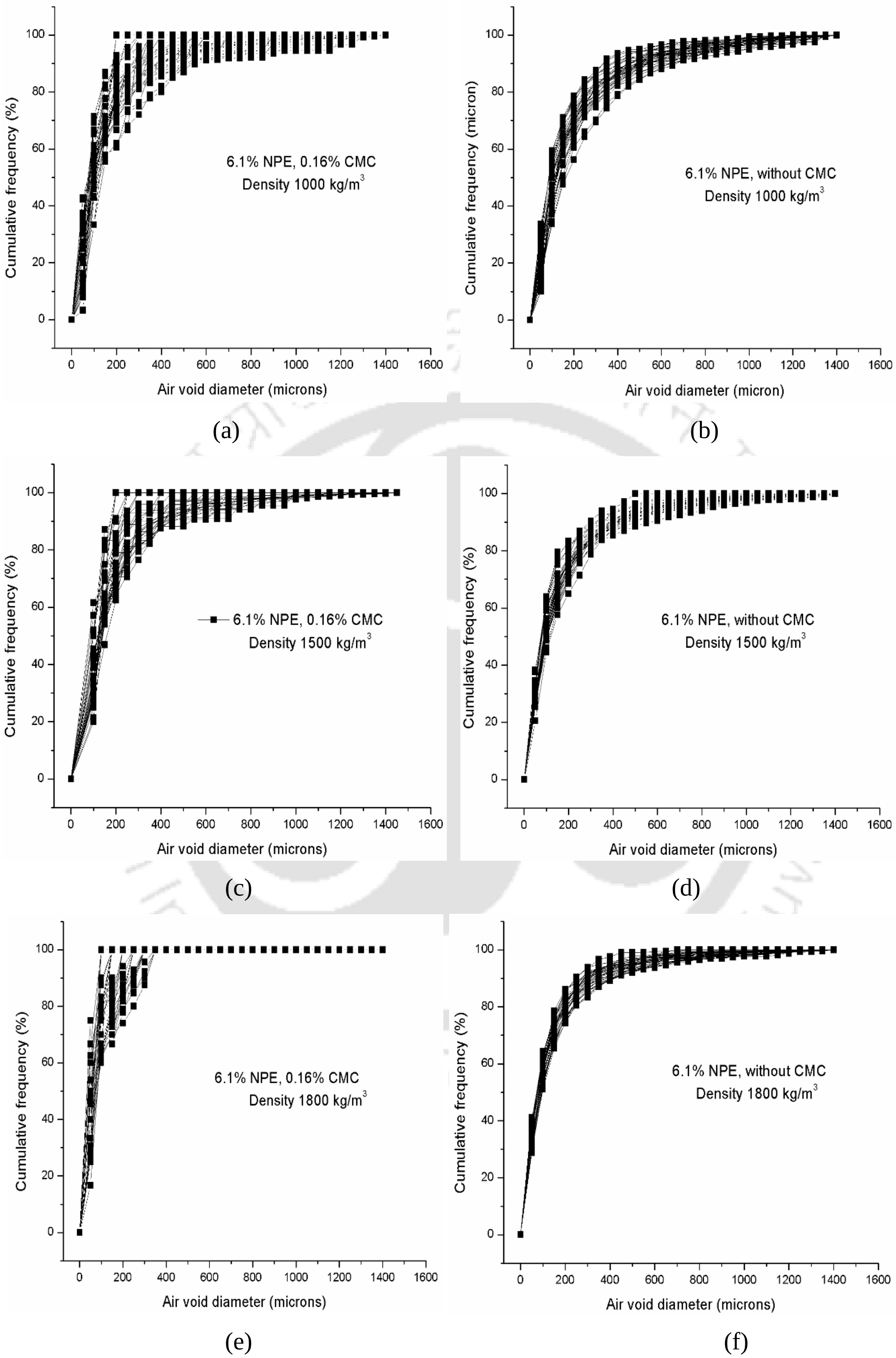


Fig. 6.8 Variation of air void size distribution with density of foam concrete (NPE)

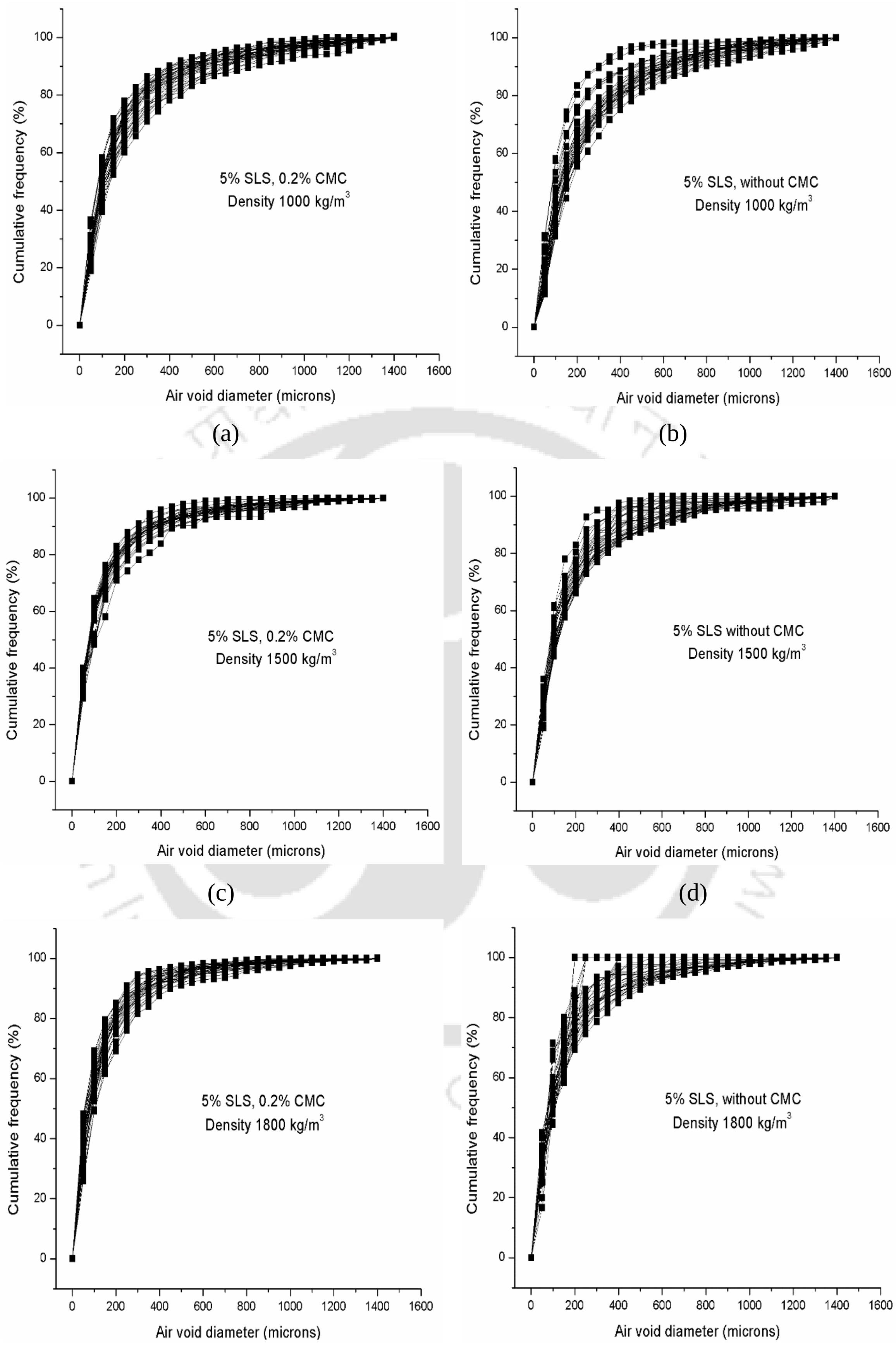
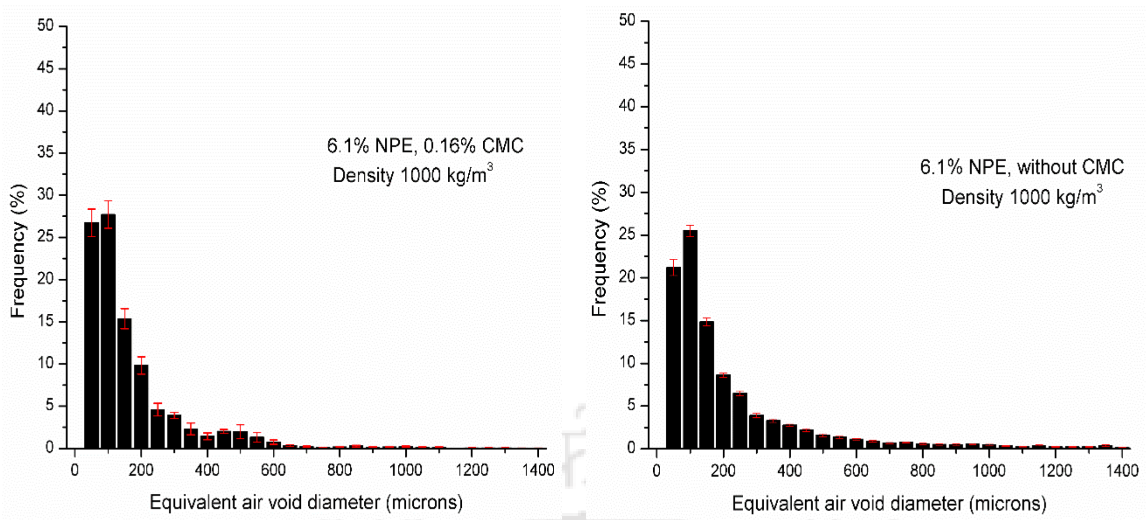
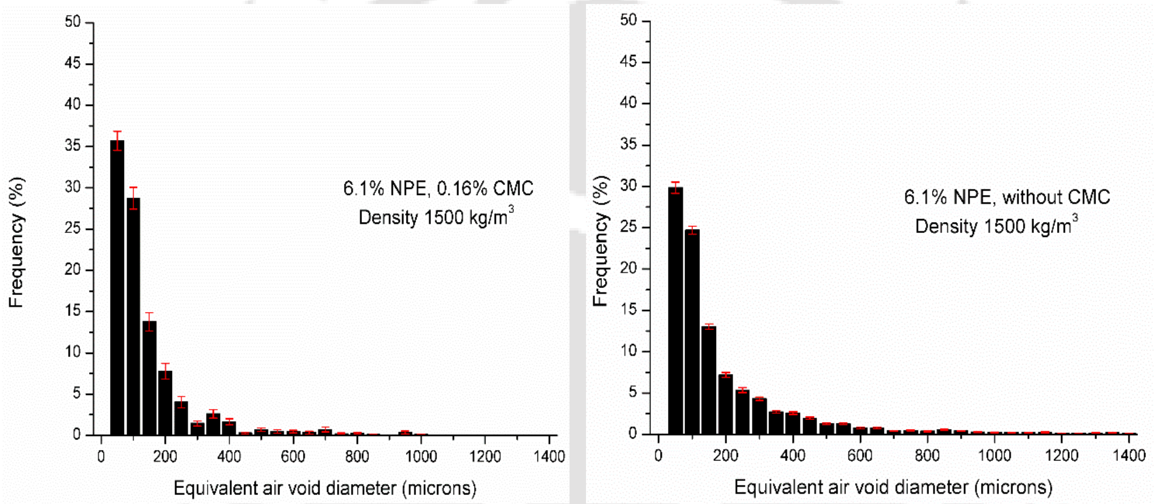


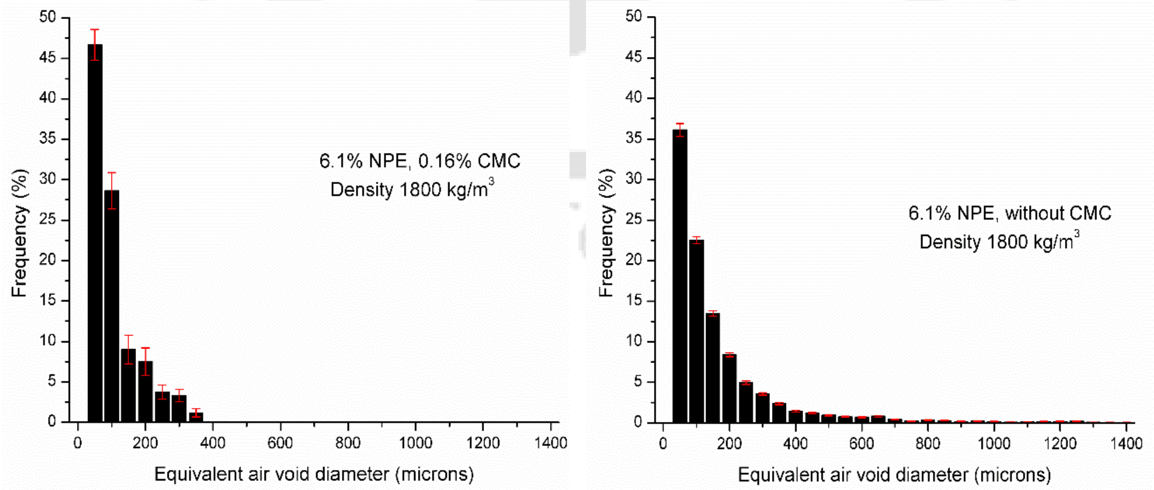
Fig. 6.9 Variation of air void size distribution with density of foam concrete (SLS)



(a) Foam concrete with fresh density 1000 kg/m^3

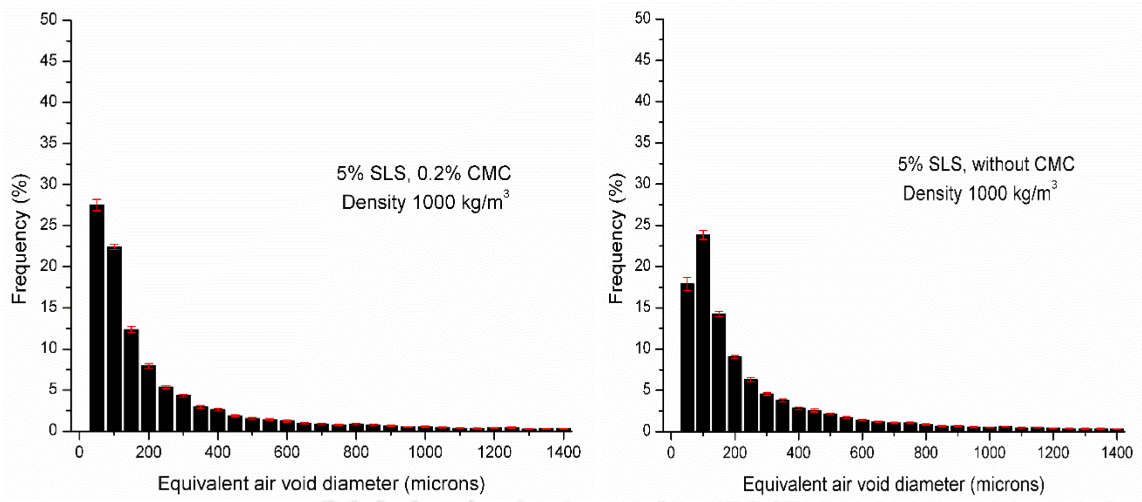


(b) Foam concrete with fresh density 1500 kg/m^3

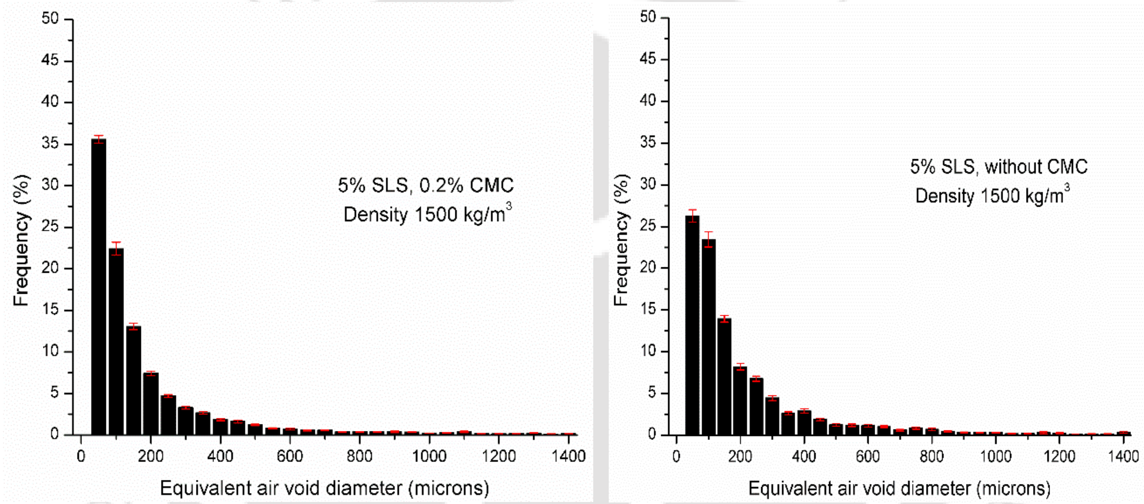


(c) Foam concrete with fresh density 1800 kg/m^3

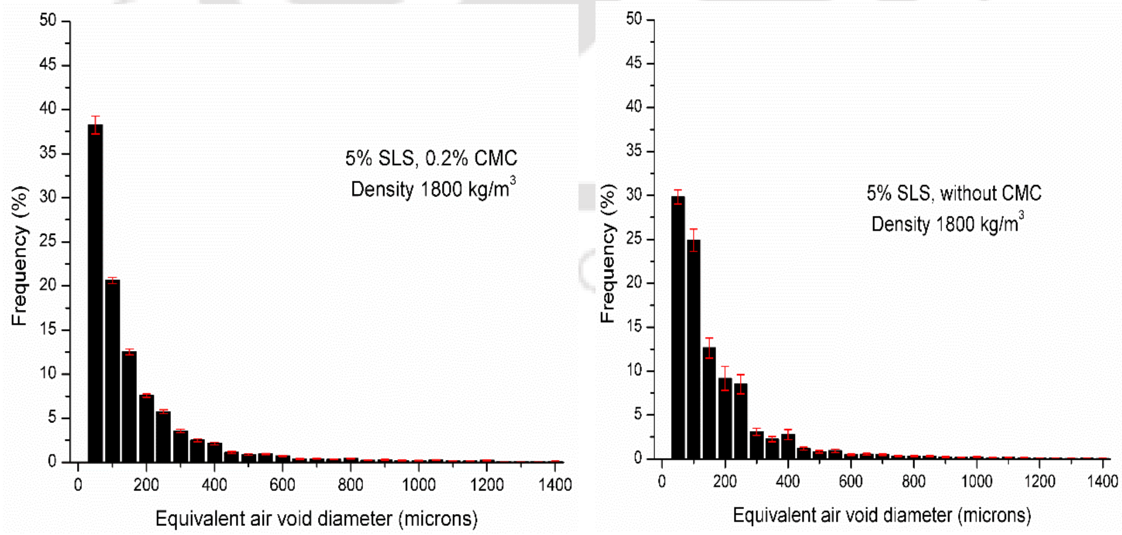
Fig. 6.10 Histogram plots showing variation of air void size distribution with density of foam concrete (NPE)



(a) Foam concrete with fresh density 1000 kg/m^3



(b) Foam concrete with fresh density 1500 kg/m^3



(c) Foam concrete with fresh density 1800 kg/m^3

Fig. 6.11 Histogram plots showing variation of air void size distribution with density of foam concrete (SLS)

Results indicates that both D50 and D90 reduces with increase in density of concrete (reduction in foam volume), but the size of larger voids reduces sharply with increase in concrete density. Hence for foam concrete with low density the merging of bubbles seem to produce larger air voids resulting in wider distribution of air void sizes.

Similar to earlier observations, the air void size distribution is relatively smaller in foam concrete with NPE when compared to that of SLS for all densities of concrete studied. Comparative analysis of microstructure of concrete with additive CMC and without additive CMC indicates that the addition of CMC to enhance the viscosity of surfactant solution results in significant improvement in microstructure of concrete with reduced air void sizes for both the surfactants. For instance a significant reduction of 40% in D90 (bigger air voids) is observed in foam concrete with design density 1800 kg/m^3, due to addition of 0.16% CMC to 6.1% NPE surfactant solution. Hence, the above observations confirms that the addition of CMC results in smaller air void microstructure, which can be attributed to enhancement of viscosity of surfactant solution and reduction in coalescence of bubbles.

Table 6.2 Air void size distribution for foam concrete with different densities (NPE)

Density (kg/m^3)	6.1% NPE, 0.16% CMC		6.1% NPE, without CMC	
	D50	D90	D50	D90
1000	126 µ (95-175 µ)	364 µ (205-570 µ)	143 µ (119-195 µ)	516 µ(367-681 µ)
1500	108 µ (81-133 µ)	299 µ (150-510 µ)	125 µ (99-150 µ)	442 µ (337-584 µ)
1800	88 µ (56-117 µ)	214 µ (120-350 µ)	114 µ (95-138 µ)	357 µ (200-448 µ)

Table 6.3 Air void size distribution for foam concrete with different densities (SLS)

Density (kg/m^3)	5% SLS, 0.2% CMC		5% SLS, without CMC	
	D50	D90	D50	D90
1000	133 µ (117-173 µ)	554 µ (260-813 µ)	162 µ (113-199 µ)	617 µ (320-827 µ)
1500	111 µ (98-149 µ)	334 µ (310-495 µ)	131 µ (112-150 µ)	481 µ (272-636 µ)
1800	97 µ (59- 137 µ)	299 µ (256-451 µ)	124 µ (96-150 µ)	383 µ (220-525 µ)

6.3.2 Circularity

Besides air void size distribution, the foam texture representing the shape of the air voids is also equally important. Moreover various researchers have reported that concrete with uniform circular air voids exhibits good mechanical and durability properties. Hence, in the present study the shape of air voids is characterized through circularity which is determined from the outer contour (perimeter) and surface area of the identified air void using the following relationship.

Circularity=4*pi*(area/perimeter^2)……………………………………………………(6.1)

A value of 1.0 indicates a perfect circle whereas the collapsed or merged bubbles can reduce the circularity value. Similar to that of air void distribution, the circularity is represented by parameters such as C 50 and C10 where C 50 represents the median value of observed circularity of air voids. In the case of circularity, irregular air voids with minimum value is going to affect the uniformity in microstructure and therefore 10% value is taken as the parameter. C10 represents the circularity of which 10% are below this value (i.e irregular air voids).

Fig. 6.12 to Fig. 6.15 shows air void circularity and the histogram plots of foam concrete produced with different concentrations of SLS and NPE surfactants. Results indicates that increase in surfactant concentration results in significant increase in circularity of air voids for foam concrete produced with both the surfactants (Table 6.4). For instance, C50, median circularity value ranges from 0.283 to 0.633 for foam concrete with 3% NPE concentration when compared to 0.475 to 0.93 for foam concrete produced with 6.1% NPE concentration. The values 0.455 and 0.766 as observed in Table 6.4 represents the overall arithmetic mean of C50 values derived for 36 images for 3% and 6.1% NPE concentrations respectively. The above improvement in circularity can be again attributed to increase in viscosity and reduction in coalescence of air bubbles at higher surfactant concentration. The results on foam bubble diameter also shows a similar trend and hence it can be concluded that foam with smaller bubble diameter leads to concrete microstructure with reduced air void sizes and improved circularity. Therefore there is a good correlation between foam bubble diameter and circularity of air voids in concrete.

Comparative studies on circularity of air voids for two surfactants indicates that the circularity of air voids are similar for both the surfactants, however C10 values are slightly higher for foam concrete with NPE. Studies on variation of circularity with density of concrete indicates that there is significant reduction in circularity of air voids with reduction in density of concrete due to increase in foam volume (Fig. 6.16 to Fig. 6.19; Table 6.5 and Table 6.6). In particular, 9 to 15% reduction in C10, is observed with reduction in concrete density from 1800 to 1000 kg/m^3. Comparative analysis of microstructure of concrete with additive CMC and without additive CMC indicates that there is significant enhancement of 40 to 59% in circularity parameters of air voids in concrete due to addition of CMC particularly at lower density of 1000 kg/m^3 (Table 6.5 and Table 6.6). However in case of higher density of concrete say 1800 kg/m^3, since the volume of foam added is already very less and hence chances of merging of bubbles is also less. Therefore there is not much significant improvement in circularity due to addition of CMC in most of the mixes of foam concrete with higher density.

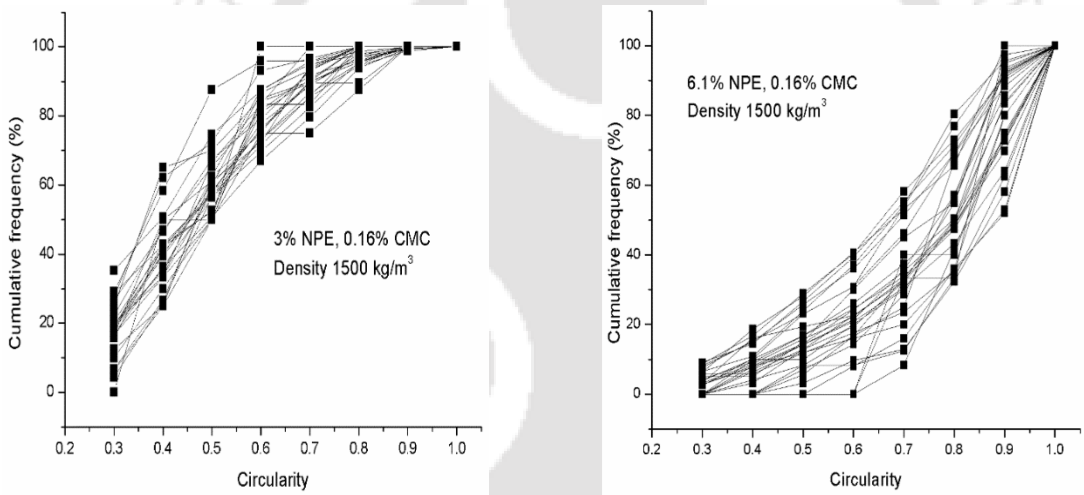


Fig. 6.12 Variation of circularity of air voids in foam concrete with surfactant concentration (NPE)

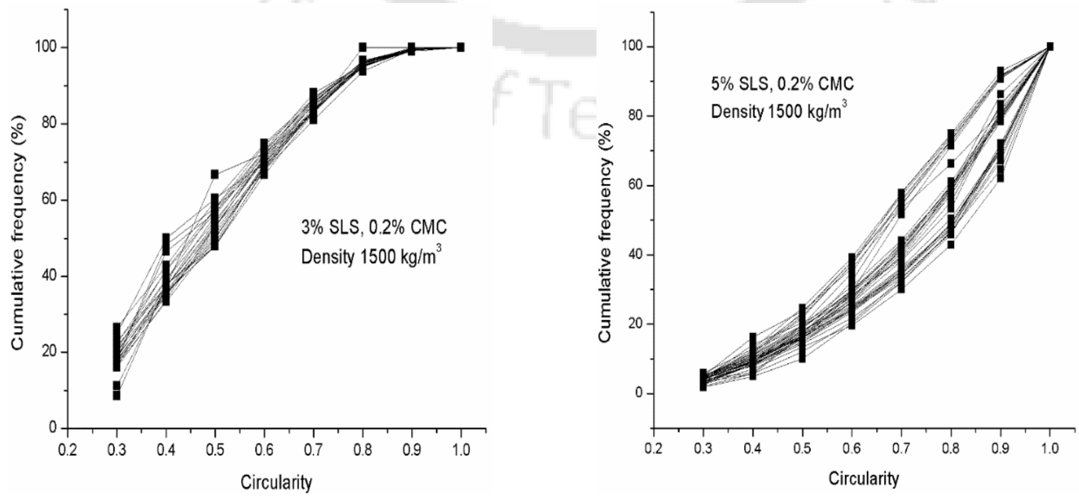


Fig. 6.13 Variation of circularity of air voids in foam concrete with surfactant concentration (SLS)

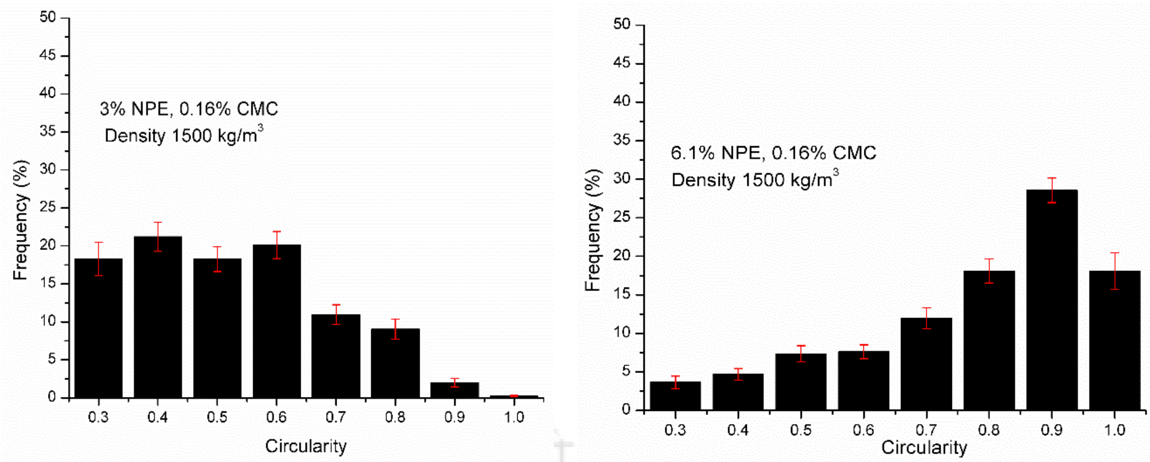


Fig. 6.14 Histogram plots showing variation in circularity of air void in foam concrete with surfactant concentration (NPE)

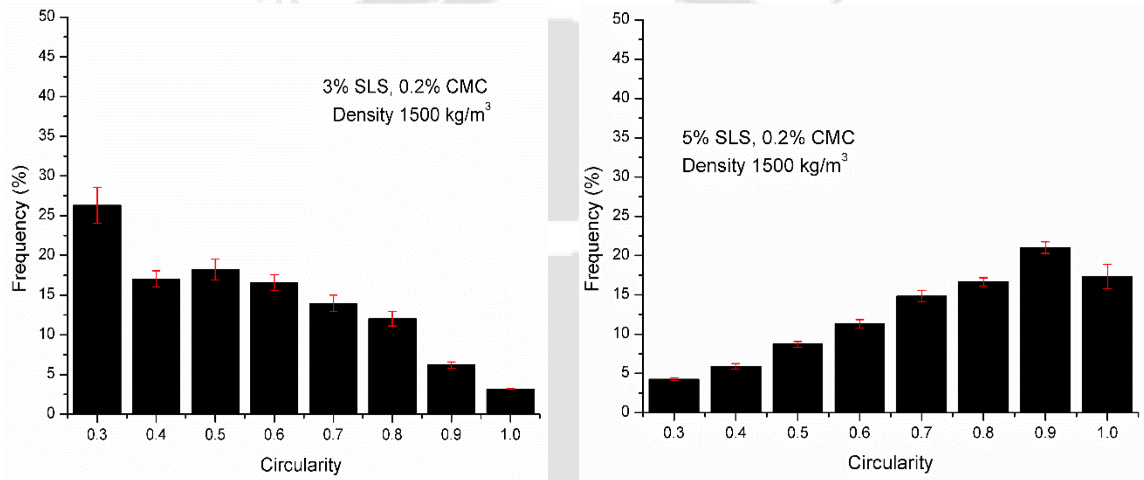


Fig. 6.15 Histogram plots showing variation in circularity of air void in foam concrete with surfactant concentration (SLS)

Table 6.4 Effect of surfactant concentration on circularity of foam concrete produced with SLS and NPE for fresh density 1500 kg/m^3

Parameters	3% NPE, 0.16% CMC	6.1% NPE, 0.16% CMC	3% SLS, 0.2% CMC	5% SLS, 0.2% CMC
C50	0.455 (0.283- 0.633)	0.766 (0.475-0.93)	0.451 (0.261- 0.588)	0.75 (0.65-0.837)
C10	0.278 (0.216-0.44)	0.468 (0.244-0.82)	0.253 (0.212- 0.301)	0.406 (0.34-0.498)

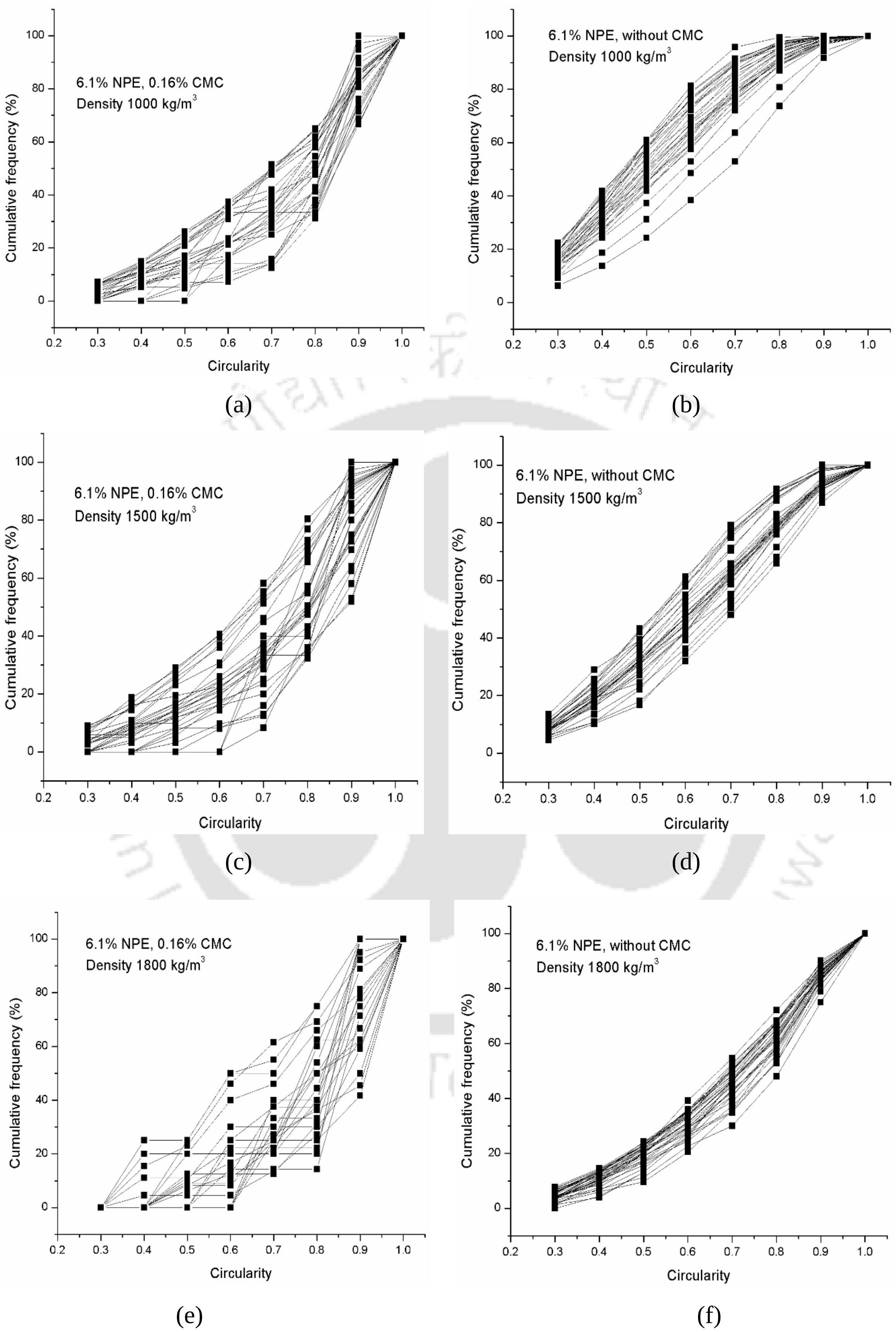


Fig. 6.16 Variation of circularity of air voids with density of foam concrete (NPE)

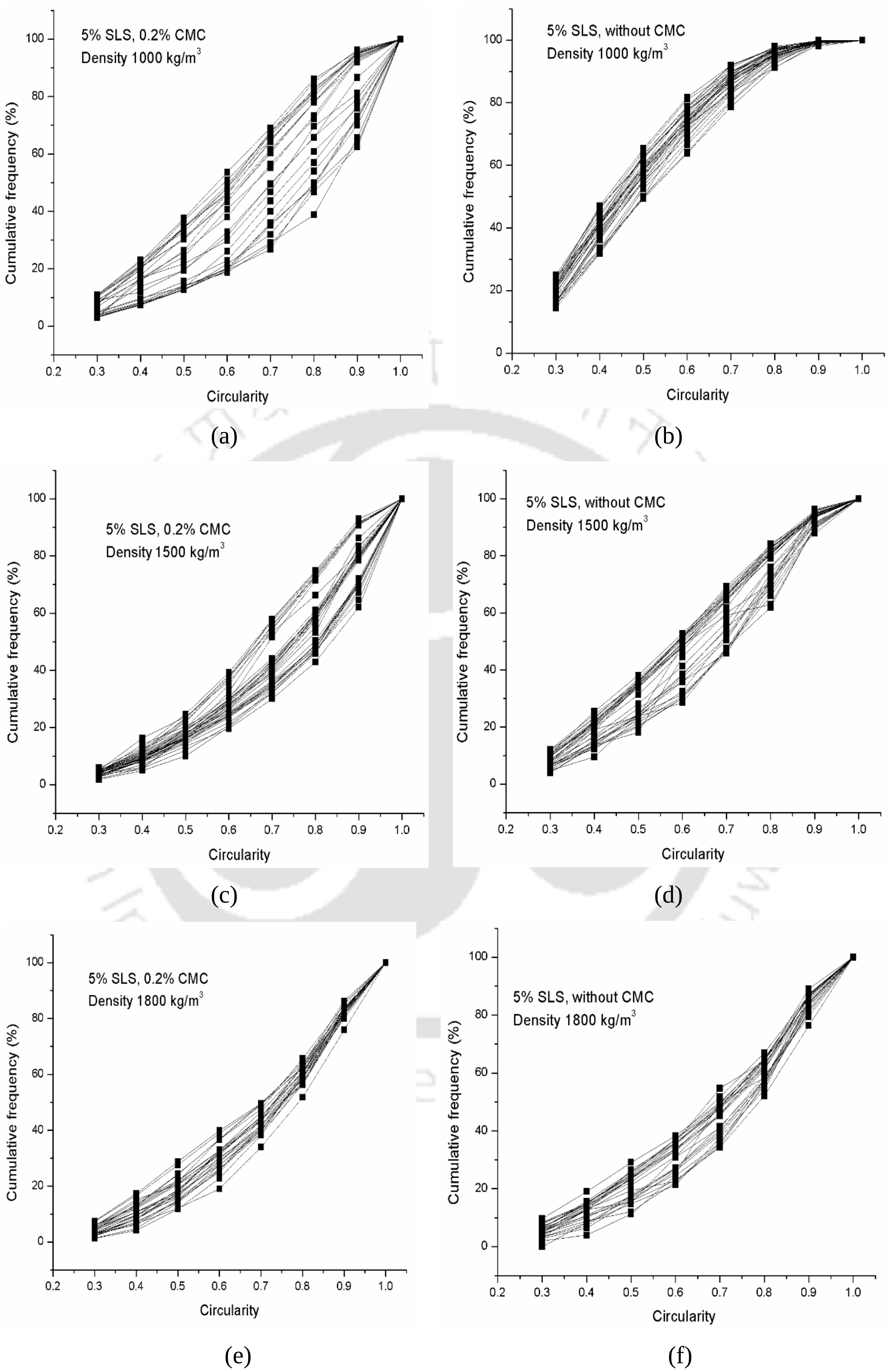
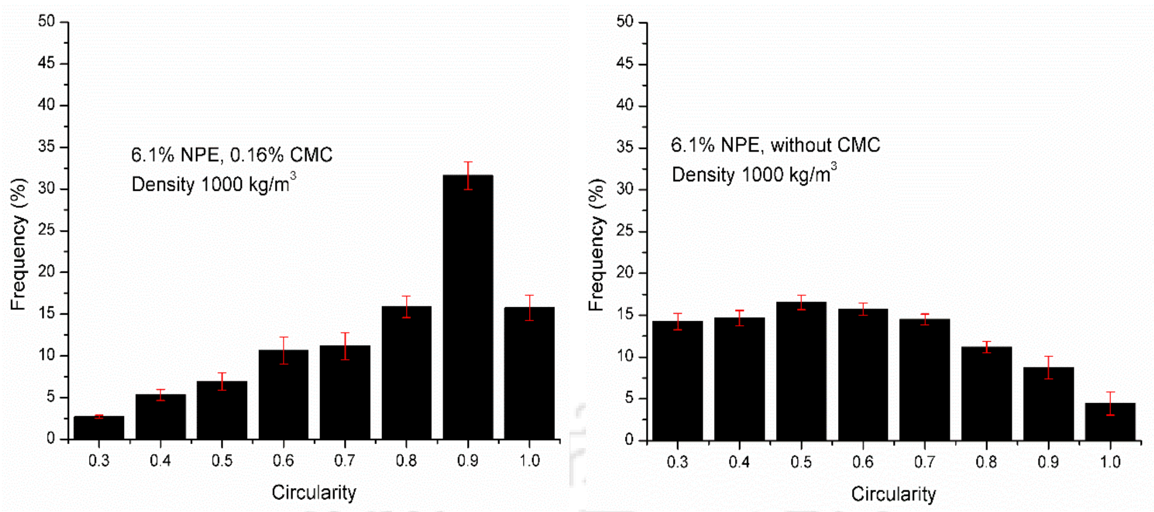
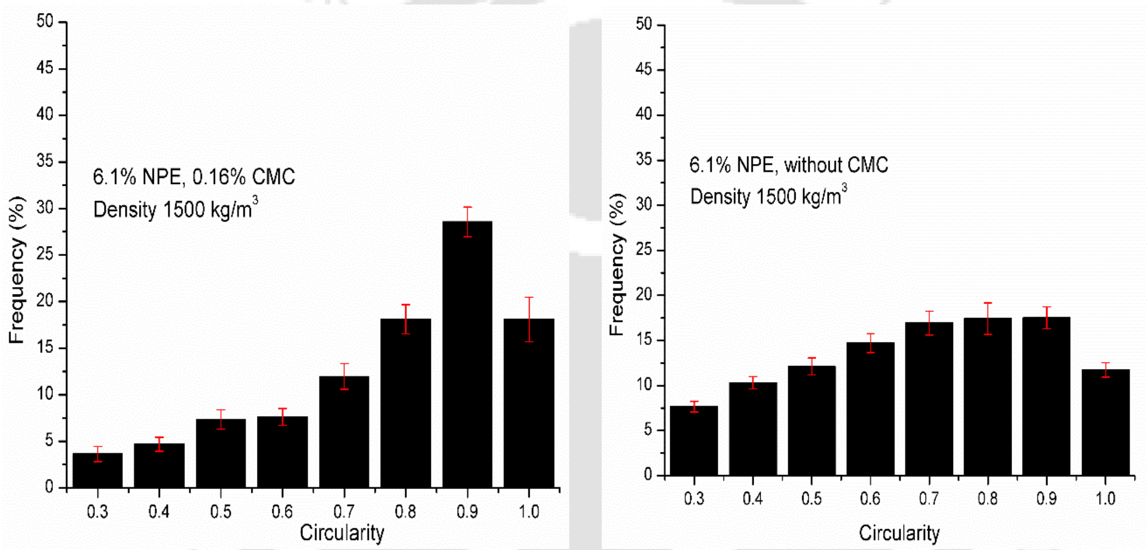


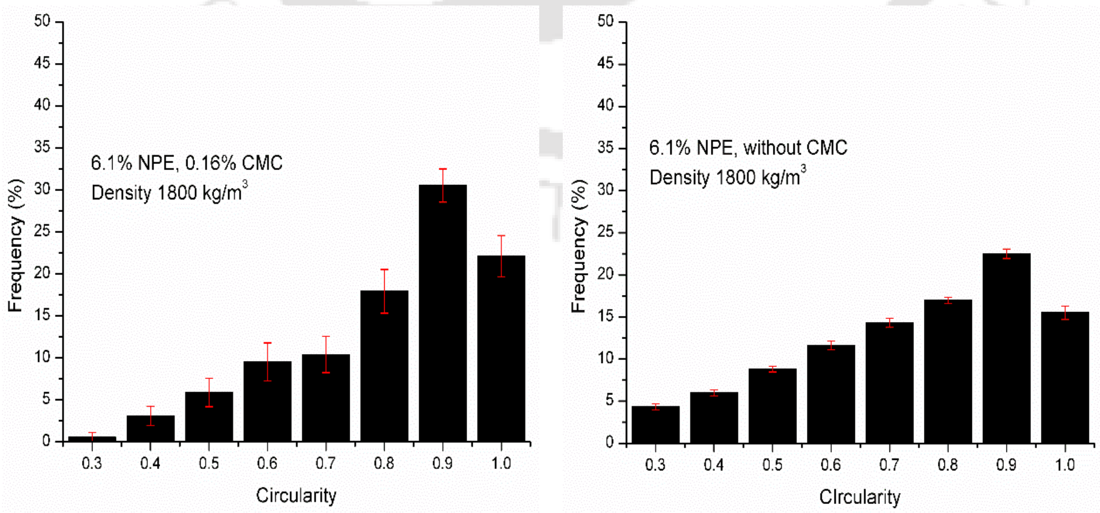
Fig. 6.17 Variation of circularity of air voids with density of foam concrete (SLS)



(a) Foam concrete with fresh density 1000 kg/m^3

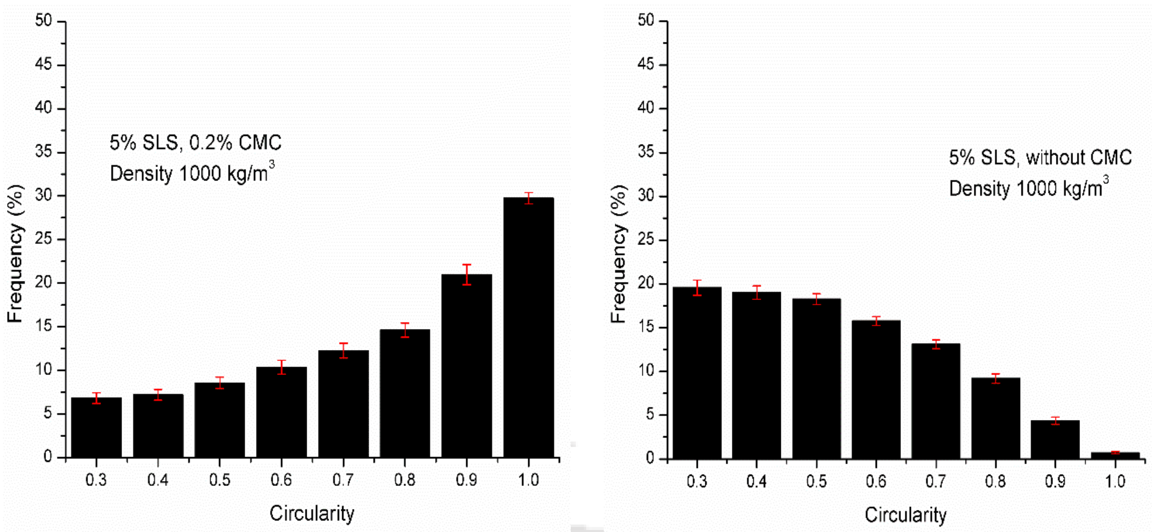


(b) Foam concrete with fresh density 1500 kg/m^3

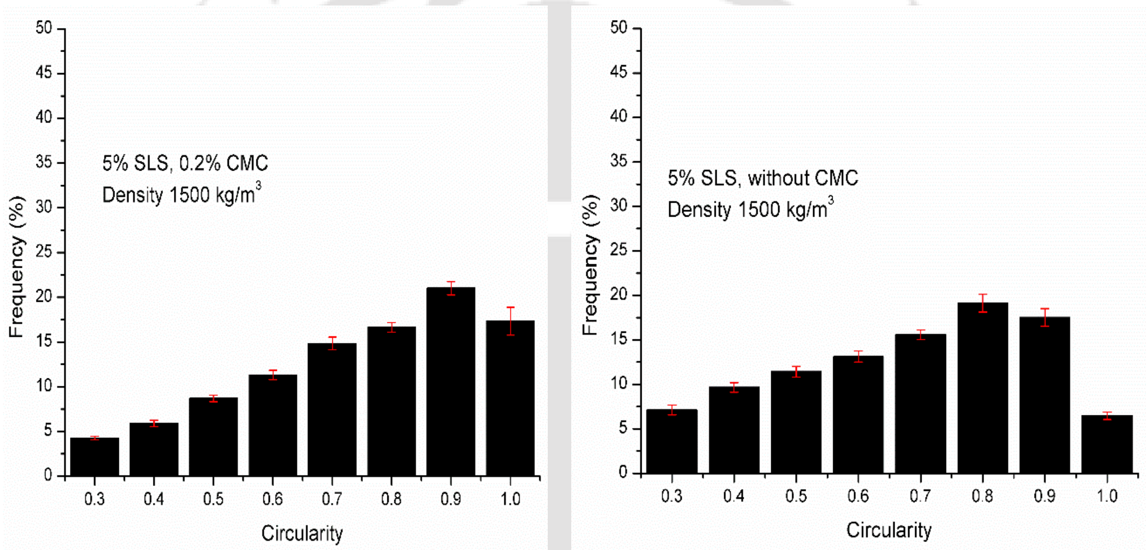


(c) Foam concrete with fresh density 1800 kg/m^3

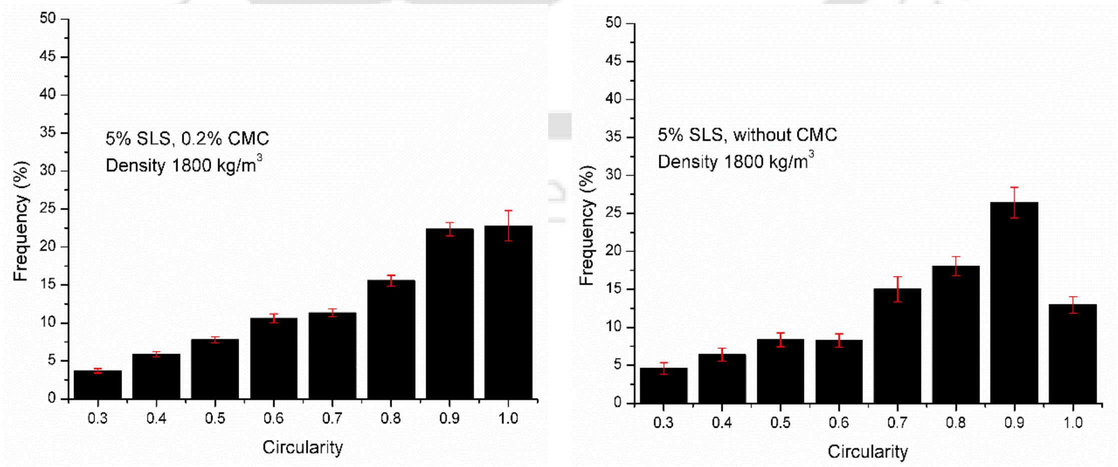
Fig. 6.18 Histogram plots showing variation of air void circularity in foam concrete with density (NPE)



(a) Foam concrete with fresh density 1000 kg/m^3



(b) Foam concrete with fresh density 1500 kg/m^3



(c) Foam concrete with fresh density 1800 kg/m^3

Fig. 6.19 Histogram plots showing variation of air void circularity in foam concrete with density (SLS)

Table 6.5 Circularity for foam concrete with surfactant NPE

Fresh density (kg/m^3)	6.1% NPE, 0.16% CMC		6.1% NPE, without CMC	
	C50	C10	C50	C10
1000	0.766 (0.53 -0.859)	0.453 (0.28-0.712)	0.544 (0.445-0.837)	0.287 (0.244-0.442)
1500	0.766 (0.516-0.9)	0.468 (0.244-0.82)	0.643 (0.507-0.866)	0.346 (0.274-0.68)
1800	0.787 (0.475-0.93)	0.532 (0.25-0.75)	0.728 (0.67-0.86)	0.394 (0.33-0.567)

Table 6.6 Circularity for foam concrete with surfactant SLS

Fresh density (kg/m^3)	5% SLS, 0.2% CMC		5% SLS, without CMC	
	C50	C10	C50	C10
1000	0.735 (0.56-0.86)	0.373 (0.26-0.47)	0.463 (0.390-0.589)	0.253 (0.235-0.3)
1500	0.75 (0.65-0.837)	0.406 (0.34-0.498)	0.653 (0.581-0.731)	0.338 (0.282-0.424)
1800	0.769 (0.702-0.88)	0.411 (0.32-0.53)	0.725 (0.4-0.825)	0.417 (0.24-0.73)

6.4 Density, Strength and Moisture Movement behaviour of Foam Concrete

6.4.1 Relationship between Fresh Density and Dry Density

The density of concrete can be measured in two phases: fresh and dry densities. Both fresh and dry densities of concrete are useful at different stages. The fresh density is to be considered for designing the mix and for casting control, while dry density controls mechanical, physical and durability properties of hardened concrete. Hence there is a need to establish the relationship between them. The dry density was measured by placing the cubes in the oven at 110°C till the cubes attained constant weight, after subjecting to 28 days of moist-curing. Fig. 6.20 exhibits a linear relationship between dry density and fresh density with an R^2 value of 0.996.

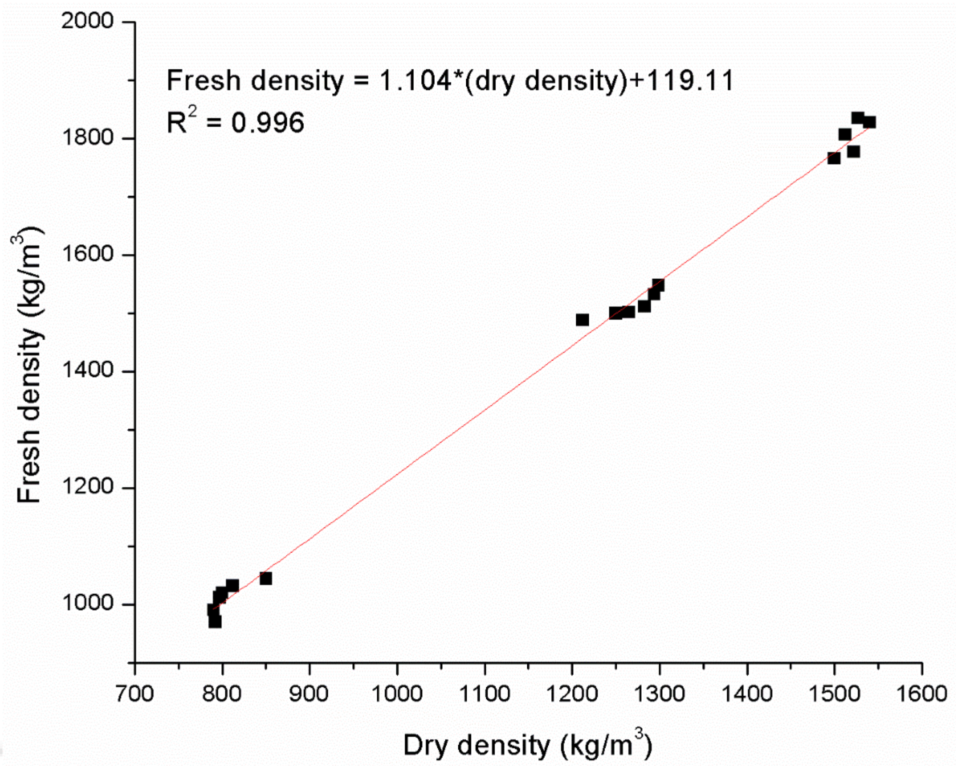


Fig. 6.20 Correlation between fresh density and dry density of foam concrete

6.4.2 Compressive Strength Behaviour

This section reports the results of studies on the influence of variation in surfactant concentration, type of surfactant, addition of additive, variation in density of concrete (foam volume) on compressive strength behavior of concrete. The correlation between density, strength and air void characteristics of concrete is also studied.

The materials and their properties and mix proportioning have been described in the earlier section 5.2.1 and 5.2.2. Foam concrete mixes of three different fresh densities viz., 1000, 1500 and 1800 kg/m^3 are made by varying the foam volume from 50% to 12%. The optimum water solids ratio derived through experimental investigations for different design densities as discussed in section 5.3.1 are adopted. For each density of foam concrete and for both the surfactants SLS and NPE, 6 cubes of size 50 mm are cast and moist cured for 28 days for measurement of strength. The compressive strength tests are carried out in accordance to ASTM C 796-97 after 28 days of water curing.

Firstly, the influence of surfactant concentration behaviour on compressive strength of concrete need to be studied. The results on compressive strength of concrete made with two different surfactants (SLS and NPE) with different surfactant concentrations are presented in Table 6.7. Results indicates that increase in surfactant concentration from 3 to 6.1% for NPE and from 3 to 5% for SLS results in significant increase of 37% and 29% in compressive strength of concrete respectively. The reason for the above improvement in compressive strength can be attributed to improvement in microstructure of concrete with increase in surfactant concentration. For instance, studies on air void characteristics indicates that increase in NPE concentration from 3 to 6.1% and increase in SLS concentration from 3 to 5% result in reduction of 24% and 44% in air void size respectively. Furthermore a significant improvement of 65% in circularity is observed with increase in surfactant concentration for both the surfactants. However it is to be noted that further increase in surfactant concentration beyond 6.1% do not result in significant improvement in compressive strength of concrete.

Having verified the optimum surfactant concentration levels from compressive strength of foam concrete perspective, as a next step influence of variation in density of concrete is studied for foam concrete mixes produced with two different surfactants SLS and NPE at optimum levels of 5% and 6.1% surfactant concentration respectively. Fig. 6.21 presents the variation of compressive strength with dry density of concrete for two surfactants SLS and NPE at optimum surfactant concentration levels. The influence of addition of additive CMC on compressive strength characteristics are also investigated. Experimentally, it is observed that compressive strength has a direct relationship with density and decreases significantly with reduction in density of concrete (increase in foam volume). This is supported by literature that reports that the strength of foam concrete is mainly influenced by volume of entrained air voids unlike normal weight concrete where w-s ratio is the predominant factor (Nambiar and Ramamurthy 2006a; Ramamurthy and Narayanan 2000; Ranjani and Ramamurthy 2008).

Table 6.7 Variation of compressive strength of foam concrete with surfactant concentration (design fresh density 1500 kg/m^3)

Name of surfactant	28 days compressive strength (MPa)
3% NPE, 0.16% CMC	4.86
6.1% NPE, 0.16% CMC	6.67
10% NPE, 0.16% CMC	6.77
3% SLS, 0.2% CMC	4.68
5% SLS, 0.2% CMC	6.06
10% SLS, 0.2% CMC	6.108

Further examination of influence of addition of CMC on compressive strength behaviour establishes that 10 to 30% enhancement in strength can observed in foam concrete produced with CMC for both the surfactants SLS and NPE. Also, the improvement in strength due to addition of CMC is more apparent in mixes with lower concrete density (higher foam volume). Possible justification for the above improvement in strength can be again attributed to the improved microstructure of concrete with reduced air void sizes in concrete mixes with CMC. Further comparative studies on influence of type of surfactant on compressive strength indicates that foam concrete produced with NPE results in 10 to 25% higher compressive strength than that of SLS (Fig. 6.21). Improved microstructure with smaller and uniform air voids in foam concrete with NPE can be attributed to its relatively higher strength.

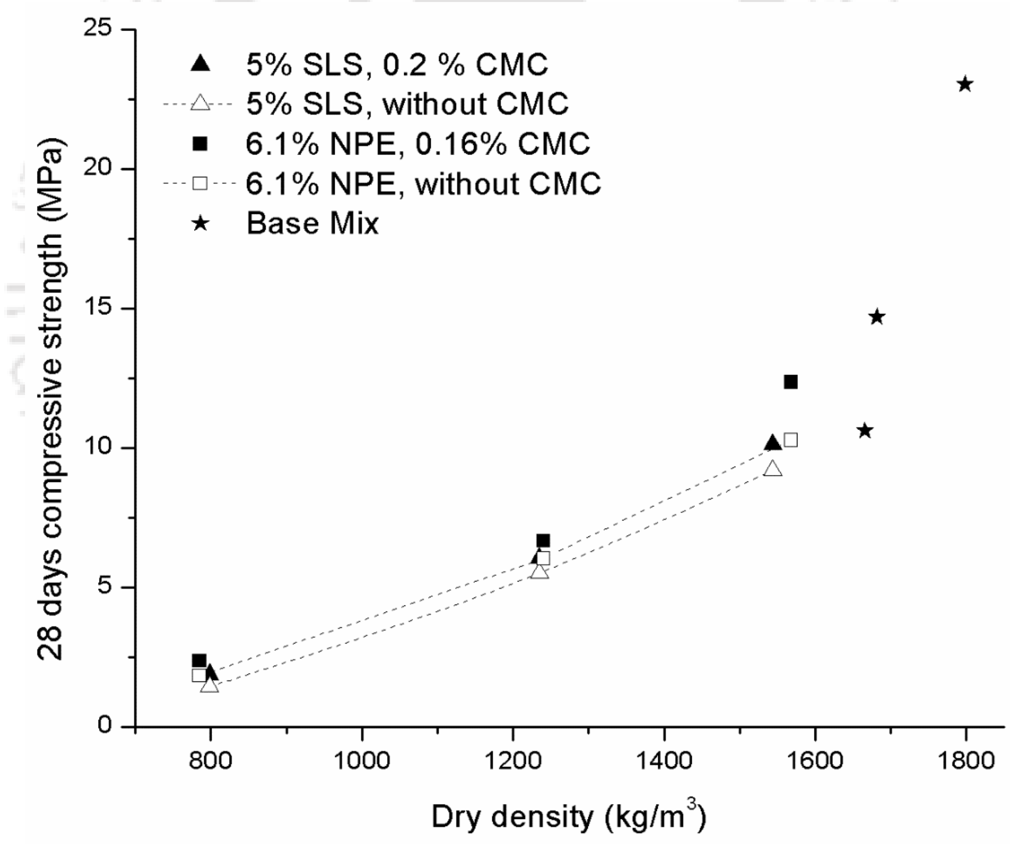


Fig. 6.21 Variation of compressive strength with dry densities

6.5 CORRELATION BETWEEN AIR VOID PARAMETERS, DENSITY AND STRENGTH OF FOAM CONCRETE

Fig. 6.22 to Fig. 6.25 represent density and strength as a function of the air-void size parameters for foam concrete produced with both the surfactants. With increase in density of concrete both the median void diameter (D50) and oversized voids (D90) becomes smaller for foam concrete mixes with both the surfactants. Also it is to be noted that the variation of D50 with density of concrete is narrower when compared to that of D90. Furthermore it is observed that D90 curves for both the surfactants converge at higher densities indicating that the voids are of similar sizes in foam concrete mixes with lesser foam volume and for mixes without CMC (Fig. 6.23). Fig. 6.24 and Fig. 6.25 shows that an increase in air void diameter leads to reduction in strength and thus the diameter and strength are inversely proportional. The variation of D50 with strength is similar for both the surfactants as the variation of D50 is narrower. Even smaller variation in median diameter (D50) results in steeper reduction in strength when compared to that of D90. The D90 distribution curves of both the surfactants tends to converge in high strength mixes (mixes with higher density) as the void size distribution is similar for both the surfactants in mixes with lesser foam volume and mixes without CMC (Fig. 6.25). However in mixes with CMC, there is some considerable difference in air void size distribution of D90 for both the surfactants even in mixes with lesser foam volume. Possible justification for this is that the addition of CMC results in significant reduction in air void size of NPE when compared to that of SLS which is more apparent even in mixes with lesser foam volume. Fig. 6. 26 to Fig. 6. 29 represent correlation between circularity parameters and density and strength of foam concrete produced with both the surfactants. With a reduction in strength and density, the circularity parameters C50 and C10 reduces.

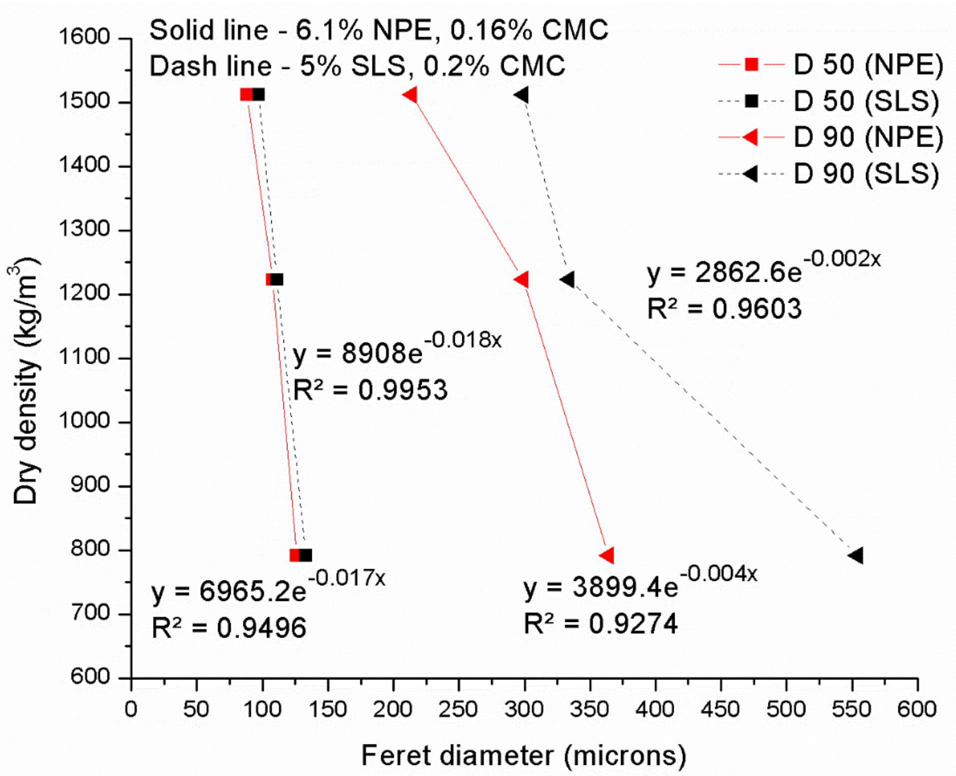


Fig. 6.22 Density Vs air void size distribution parameters (with CMC)

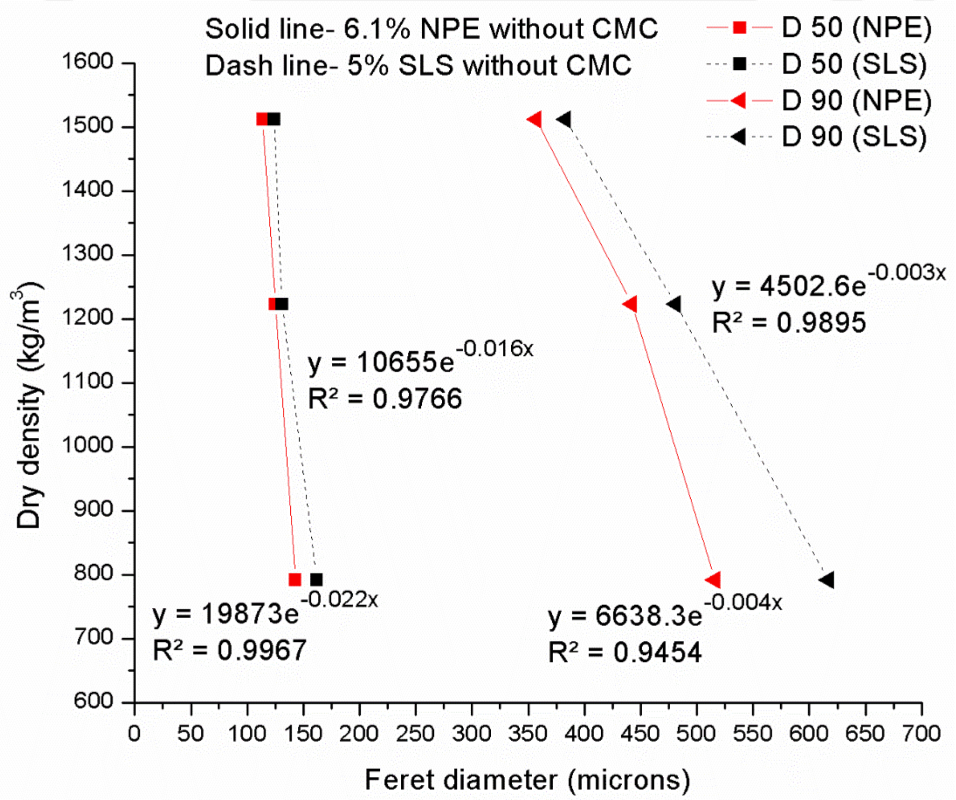


Fig. 6.23 Density Vs air void size distribution parameters (without CMC)

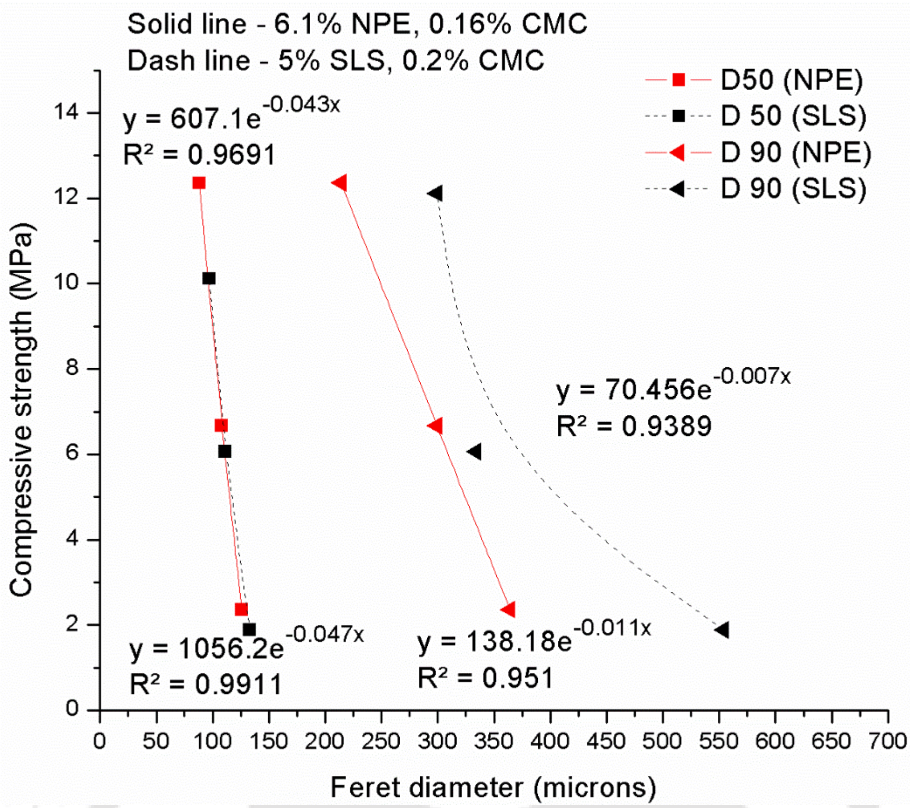


Fig. 6.24 Strength Vs air void size distribution parameters (with CMC)

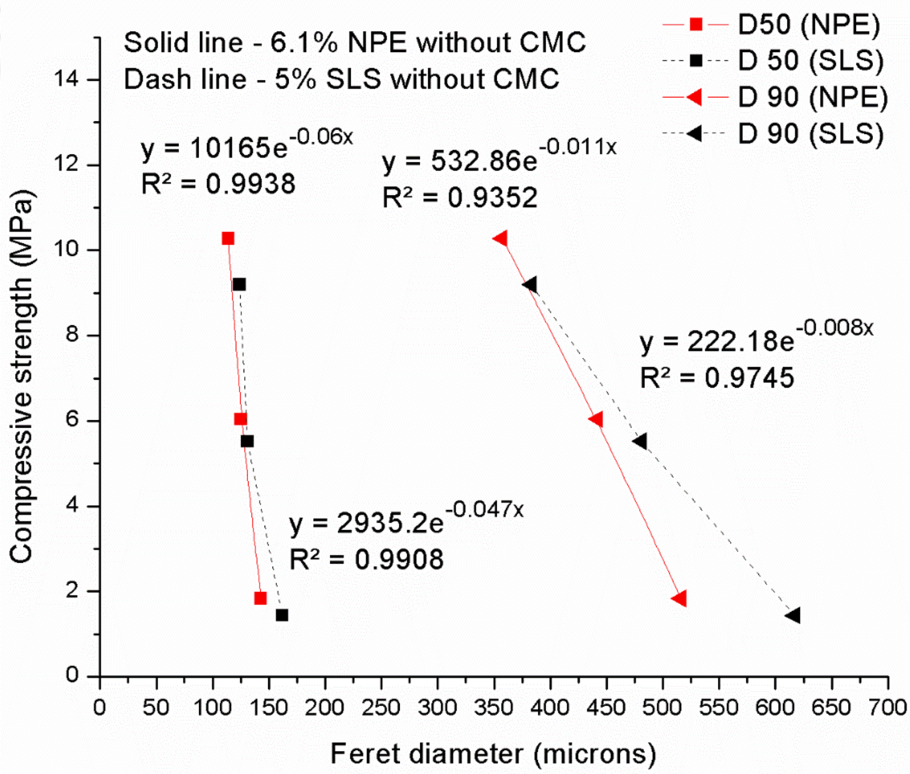


Fig. 6.25 Strength Vs air void size distribution parameters (without CMC)

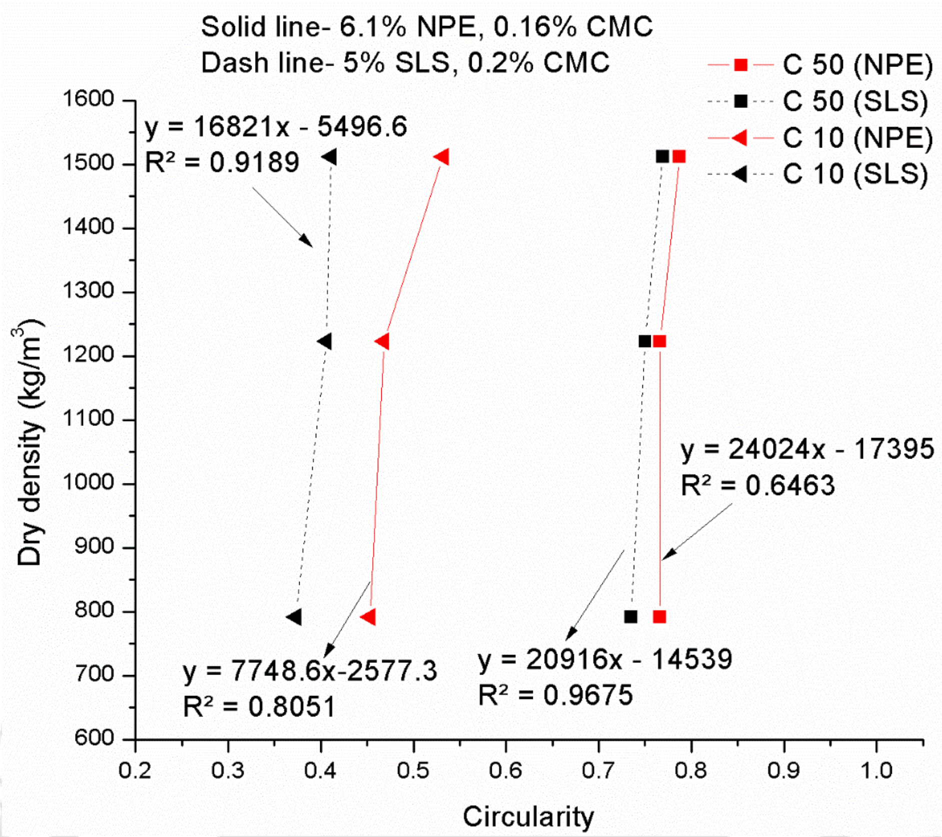


Fig. 6.26 Density Vs circularity of air void (with CMC)

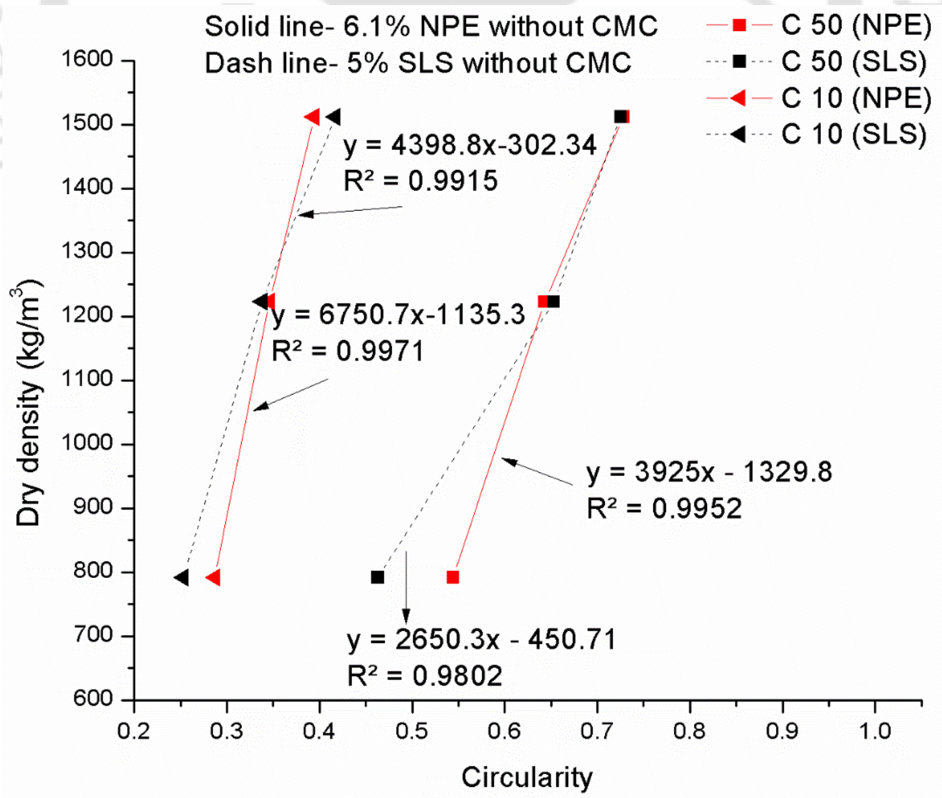


Fig. 6.27 Density Vs circularity of air void (without CMC)

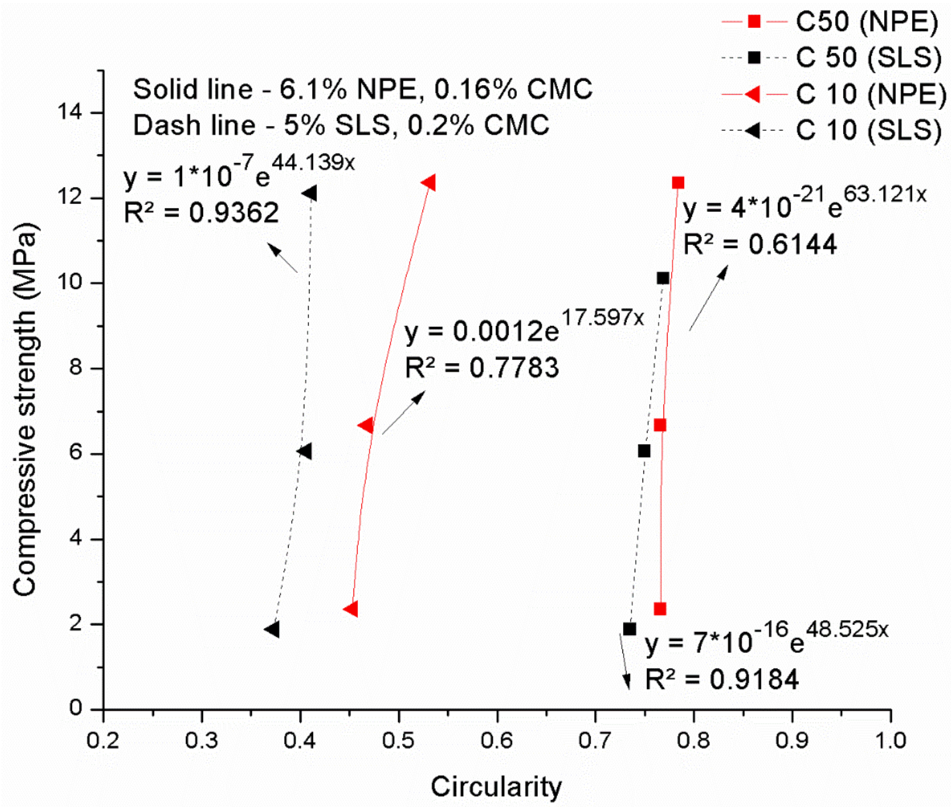


Fig. 6.28 Strength Vs circularity of air void (with CMC)

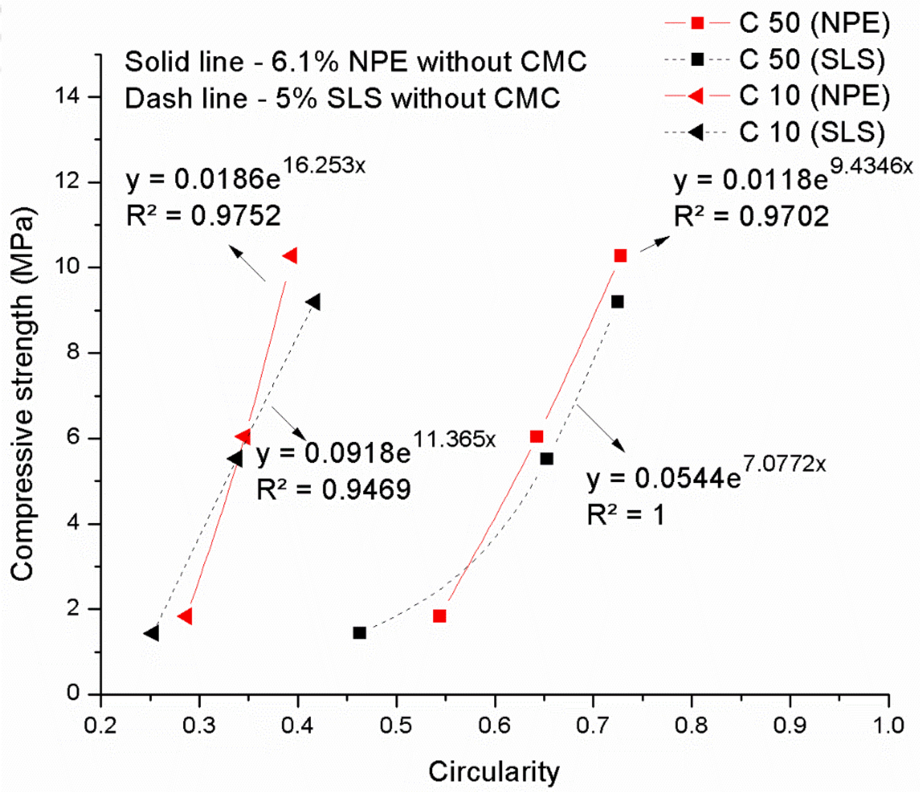


Fig. 6.29 Strength Vs circularity of air void (without CMC)

6.5.1 Moisture Movement Characteristics of Concrete

The transport of aggressive liquids into concrete depends on its permeation characteristics such as permeability, water absorption and sorption which has important bearing on durability of structure. The water absorption which is the fundamental measurement to assess the water permeation depends on the total volume of pores in concrete. There are different procedures reported in literature for measurement of water absorption such as 24 hours immersion in water (ASTM C 796), immersion till constant mass (ASTM C 642) and vacuum saturation method (ASTM C 1202) resulting in widely different results. Further it is reported that as there are significant differences in density of foam concrete , expressing water absorption as % of dry mass leads to misleading results and it is thus better to express water absorption in kg/m^3 (Kearsley and Wainwright 2001; Nambiar and Ramamurthy 2006; Ramamurthy et al. 2009)

Sorptivity which is an index of moisture movement into unsaturated specimens has emerged as an important index of concrete durability because the test method reflects the way that most concrete will be penetrated by water and other agents (Dias 2004). A number of research works in normal weight concrete have studied the effect of variation of sorptivity with variation in strength, specimen preconditioning, curing method and addition of mineral admixtures. Limited sorptivity studies have also been made on foam concrete for the influences of foam volume (Giannakou and Jones 2002; Madjoudj et al. 2002; Nambiar and Ramamurthy 2007a), filler type and fly ash replacement for fine aggregate (Jones and McCarthy 2005a). For instance, it has been observed that replacement of cement-sand mixes with cement-sand-fly ash mixes increased the water absorption and sorptivity because of its higher water-solids ratio requirement. Further it is also reported that water movement into foam concrete is more complex due to presence of larger volume of air voids and it depends on many parameters such as porosity, pore size distribution and tortuosity (Nambiar and Ramamurthy 2007a).

Hence this section is aimed at the assessment of influence of variation in surfactant concentration, type of surfactant, addition of additive, variation in density of concrete (foam volume) on water absorption and sorption characteristics of concrete. Foam concrete mixes of three different fresh densities viz., 1000, 1500 and 1800 kg/m^3 are made by varying the foam volume from 50% to 12% using the mix proportions mentioned in

Table 5.1 in chapter 5. The optimum water solids ratio derived through experimental investigations for different design densities as discussed in section 5.3.1 are adopted.

6.5.1.1 Water Absorption

For measurement of water absorption, 6 cubes of size 50 mm are cast for each mix and moist cured for 28 days. Absorption is measured by the increase in weight of oven-dried specimens (to a constant mass) after immersion in water for 24 hours. As there is significant variation in the density of foam concrete, the water absorption is expressed as percentage of volume of concrete.

In line with earlier investigations, the influence of surfactant concentration on the water absorption characteristics is studied. The results on water absorption (% by volume) of concrete made with two different surfactants with different concentrations are presented in Table 6.8. Results indicates that increase in surfactant concentration from 3 to 10% results in 5 to 10% reduction in water absorption of concrete. Fig. 6.30 presents the variation of water absorption with dry density of concrete for two surfactants at their optimum surfactant concentration levels along with their corresponding base mixes (mortar without foam). Foam concrete produced with both the surfactants exhibits lower water absorption than the corresponding base mixes.

Table 6.8 Effect of surfactant concentration on volume of water absorbed for foam concrete produced with SLS and NPE for fresh density 1500 kg/m^3

Name of surfactant	Water absorption (% by volume)
3% NPE, 0.16% CMC	26.27
6.1% NPE, 0.16% CMC	24.44
10% NPE, 0.16% CMC	23.54
3% SLS, 0.2% CMC	26.77
5% SLS, 0.2% CMC	26.06
10% SLS, 0.2% CMC	25.41

On further examination of influence of density on water absorption, it is revealed that both the surfactants shows different trend. The water absorption of foam concrete with SLS is almost similar for all densities of concrete with slight decrease with reduction in density. However for NPE, a significant increase of 42% in water absorption is observed when concrete density is varied from 1800 to 1000 kg/m^3. Higher water absorption in mixes with higher foam volume in NPE, can be ascribed to presence of more interconnected air voids as verified through measurement of sorptivity which will be presented in the upcoming section. The above observations are similar to findings of other researchers who had reported increase in water absorption with increase in foam volume (Panesar 2013; Siva et al. 2017).

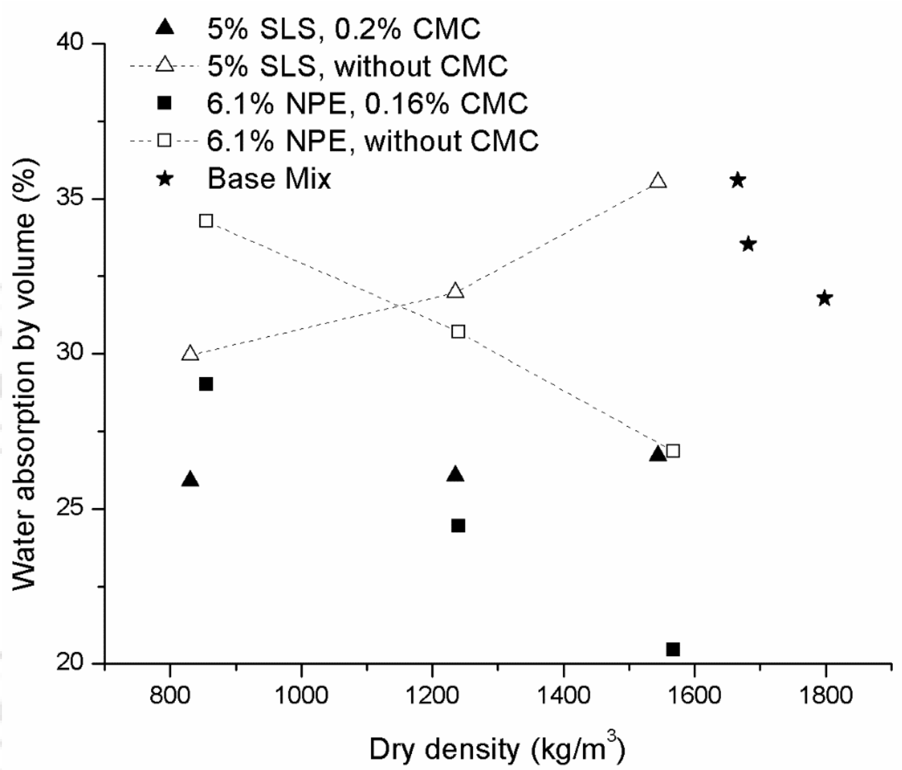


Fig. 6.30 Effect of air void size on water absorption of foam concrete with different densities

The closer examination of the influence of type of surfactant reveals that the water absorption of foam concrete mixes with NPE is lesser than that of SLS for most of the mixes studied with the exception of 1000kg/m^3 density of concrete as explained above. Furthermore studies on influence of addition of CMC on water absorption behaviour of concrete reveals that 14 to 25% reduction in water absorption is observed in concrete mixes produced with CMC. This can be ascribed to improved microstructure of concrete due to addition of CMC.

6.5.1.2 Sorptivity

For sorptivity test six cubes of size 70.7X70.7X70.7mm are cast for each mix and the specimens are demoulded after 24 hours. Water sorptivity is measured according to the method proposed by Hall (1989). The experiments are carried out under ambient conditions and the measured temperature and relative humidity are in the range of 26 to 30^{0}C and 70-85% respectively. Measurements of capillary sorption are carried out using specimens preconditioned in the oven at about 50 ± 2^0 C until constant for 3 days. Test specimens are exposed to the water on the plane of 70.7x70.7mm by placing it in a pan. The water level in the pan is maintained at about 5mm, above the base of the specimens during this experiment. The lower areas on the sides of the specimens are coated with epoxy to achieve unidirectional flow. At certain times the mass of the specimens are measured using a balance, then the amount of water absorbed is calculated and normalized with respect to the cross sectional area of the specimens exposed to the water at various times such as 4,9,16,25,36,49,….2025 min. Sorptivity(s) is obtained as the slope of the linear portion of the line of graph of cumulative water absorption per unit area of the inflow surface (i measured in mm) against square root of times (t measured in minutes) where i is calculated from i = Δw/Axρ, where Δw= increase in weight with time (gm), A= cross sectional area (mm^2), ρ= density of water (1000 kg/m^3).

The effect of density on sorption characteristics of foam concrete and base mixes for the period of 33.75 hours is studied. Table 6.9 shows the sorptivity and intercept data obtained from the sorptivity equations representing the linear portion of plots in Fig. 6.31 to Fig. 6.36 for a period of 15 hours. Foam concrete produced with both the surfactants exhibits significantly lower sorptivity than the corresponding base mixes (mortar without foam). On further examination of influence of density on sorptivity, it is revealed that both the surfactants shows different trend. The sorptivity of foam concrete with SLS shows significant decrease of 42% when foam concrete density is reduced from 1800 to 1000 kg/m^3. The decrease in sorptivity in lower density foam concrete mixes made with SLS can be attributed to reduction in volume of capillary pores due to reduction in paste content. This indicates that the entrained air voids do not contribute much to sorptivity as most of them are not interconnected. Similar observation was reported by Wong et al. 2011. However sorptivity of foam concrete with NPE shows different trend with variation in density of concrete. Similar to observations on water absorption, the NPE foam concrete mixes with density of 1000 kg/m^3 (with higher foam volume) shows a higher sorptivity value unlike SLS concrete mixes, which can be ascribed to interconnectivity between air voids.

Table 6.9 Sorptivity of foam concrete (based on quantity of water absorbed)

Fresh density	Foam volume	Surfactant	Sorptivity mm/(min)$^{0.5}$	Intercept	R^2
1000	50	6.1% NPE, 0.16% CMC	0.298	2.177	0.992
1500	25		0.199	0.445	0.998
1800	12		0.154	0.291	0.997
1000	50	6.1% NPE, without CMC	0.435	2.007	0.997
1500	25		0.354	0.893	0.991
1800	12		0.285	0.858	0.994
1000	49	5% SLS, 0.2% CMC	0.161	0.003	0.993
1500	24		0.271	-0.495	0.996
1800	12		0.277	-0.283	0.995
1000	50	5% SLS, without CMC	0.205	0.059	0.990
1500	25		0.372	0.589	0.994
1800	12		0.453	-1.695	0.988
1921	NA	Base -1 (without foam)	0.724	-1.282	0.996
1971	NA	Base -2 (without foam)	0.620	-1.854	0.989
2044	NA	Base -3 (without foam)	0.546	-1.324	0.992

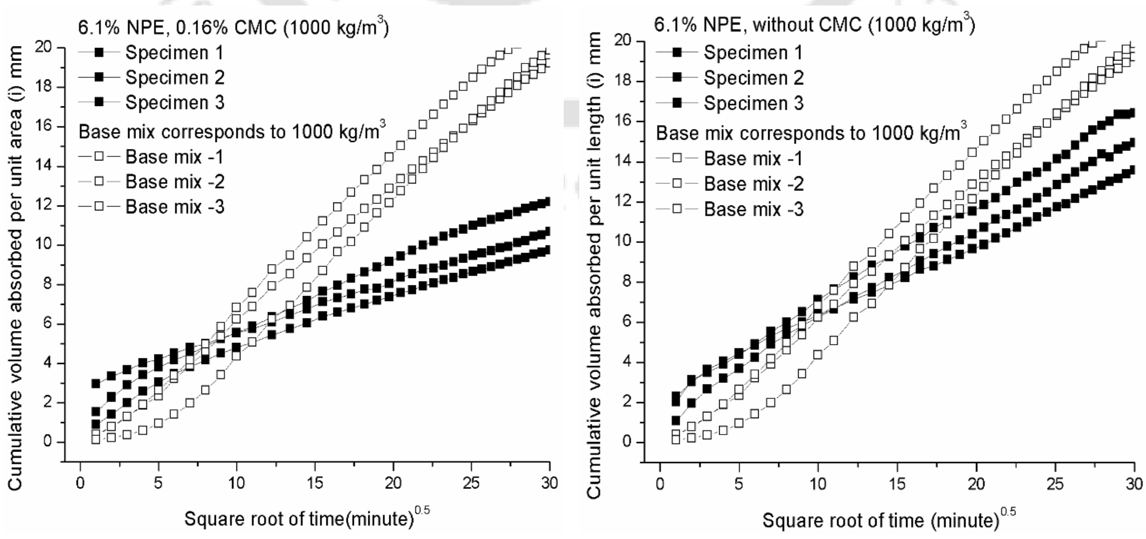


Fig. 6.31 Effect of curing on cumulative sorption of foam concrete of fresh density 1000 kg/m^3 (NPE)

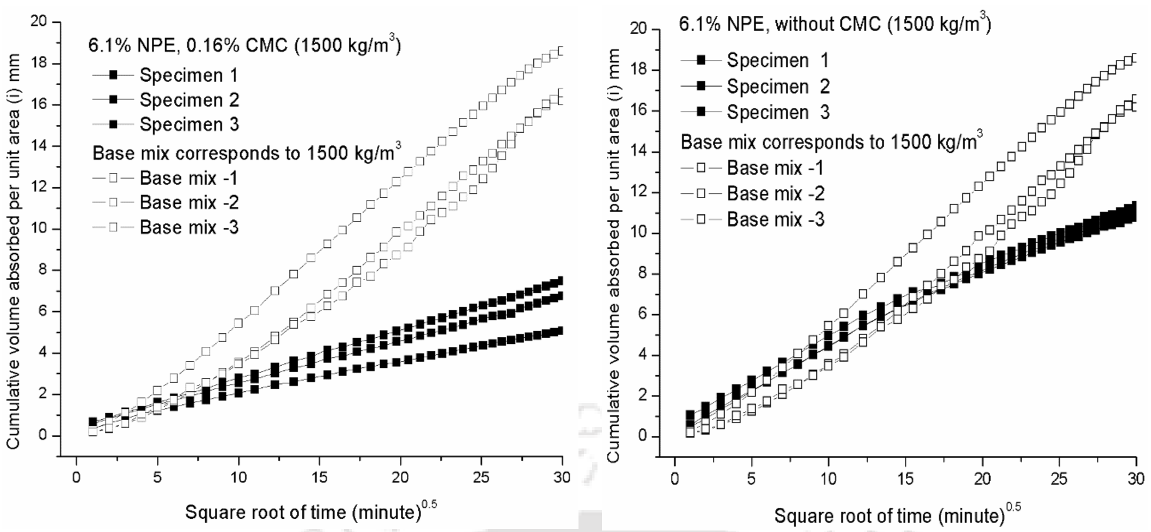


Fig. 6.32 Effect of curing on cumulative sorption of foam concrete of fresh density 1500 kg/m^3 (NPE)

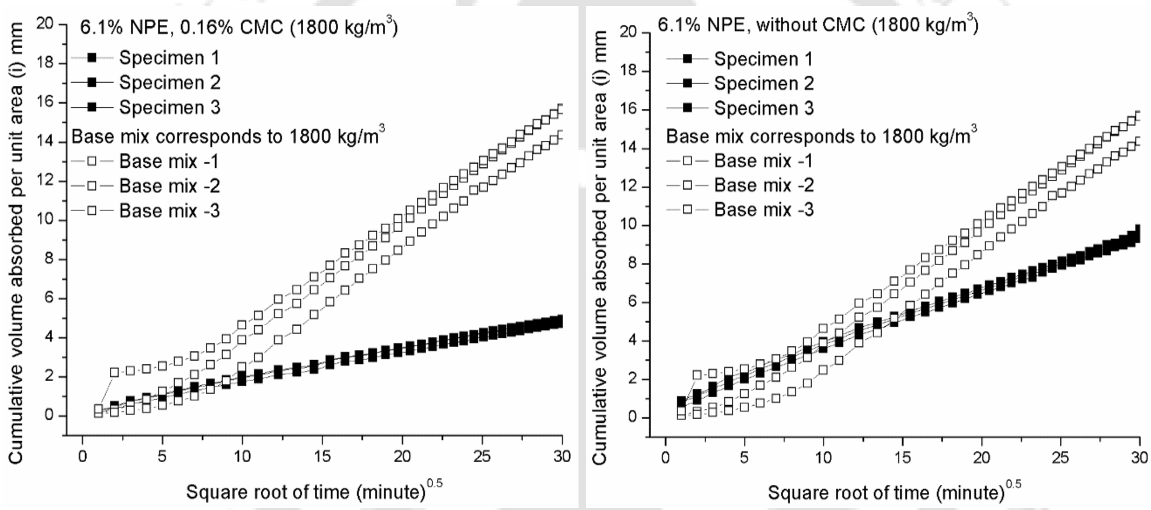


Fig. 6.33 Effect of curing on cumulative sorption of foam concrete of fresh density 1800 kg/m^3 (NPE)

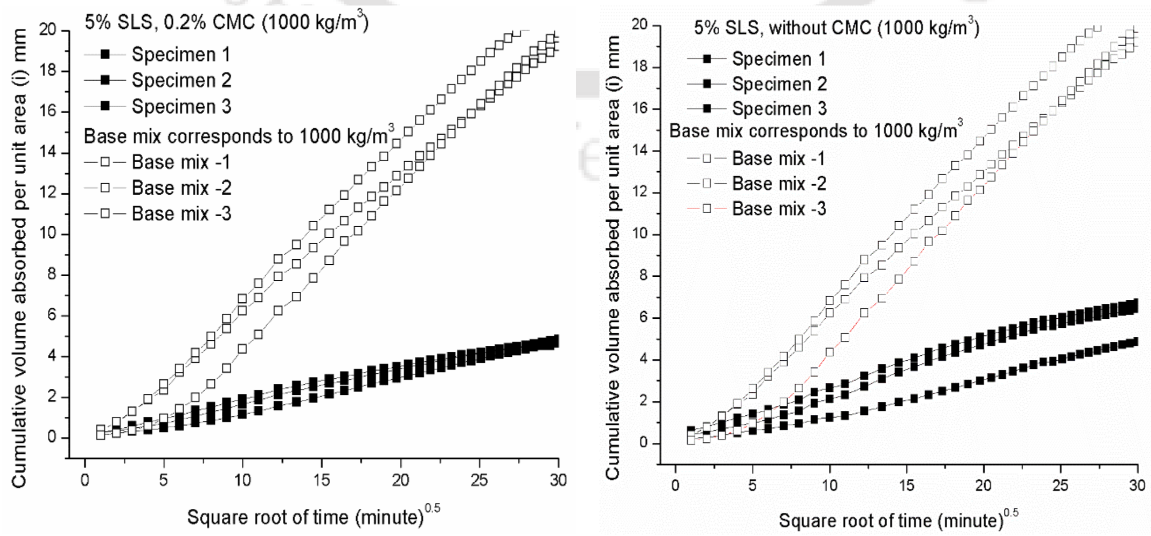


Fig. 6.34 Effect of curing on cumulative sorption of foam concrete of fresh density 1000 kg/m^3 (SLS)

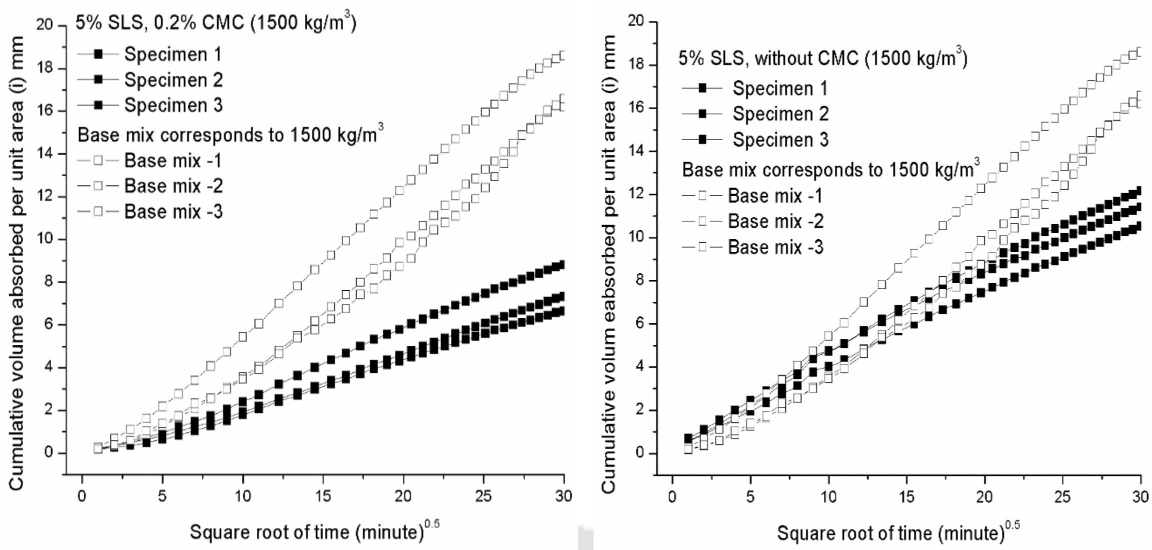


Fig. 6.35 Effect of curing on cumulative sorption of foam concrete of fresh density 1500 kg/m^3 (SLS)

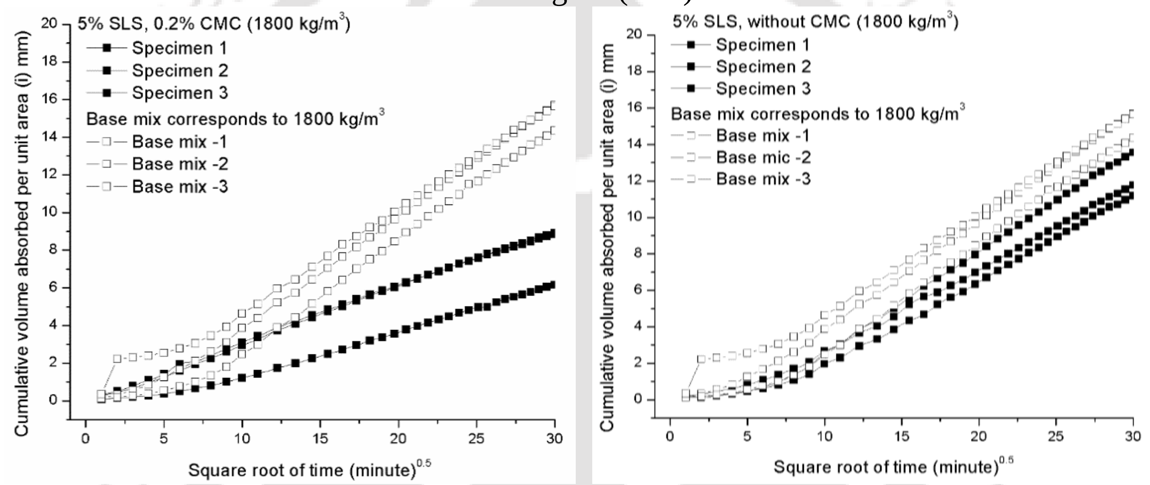


Fig. 6.36 Effect of curing on cumulative sorption of foam concrete of fresh density 1800 kg/m^3 (SLS)

Comparative studies on type of surfactant reveals that the sorptivity of foam concrete mixes with NPE is 26 to 45% lesser than that of SLS for most of the mixes studied with the exception of 1000 kg/m^3 density of concrete as explained above. Hence though the air void sizes are smaller in foam concrete with density 1000 kg/m^3 in NPE than that of SLS, however the connectivity between air voids seems to be relatively higher in mixes with higher foam volume in NPE leading to higher sorptivity values.

Furthermore studies on influence of addition of CMC on sorption behaviour of concrete shows that 21 to 45% reduction in sorptivity is observed in concrete mixes produced with CMC. This can be ascribed to improved microstructure of concrete due to addition of CMC.

6.6 SUMMARY

The air void parameters of foam concrete such as air void size distribution and circularity has been characterized through image analysis and the influence of these air void parameters on density, strength and water permeation characteristics of foam concrete are studied. Initially the effect of surfactant concentration on air void size distribution and circularity is studied. Based on the experimental results, it is noticed that increase in surfactant concentration results in significant decrease in air void size with improved circularity and hence leads to uniform microstructure of concrete with enhanced strength. As a next step, the influence of addition of CMC and variation of density of concrete on the microstructure of concrete at optimum surfactant and additive concentration is examined. The median air void size (D50) varies from 88 to 126 microns and 97 to 133 microns for foam concrete with fresh density ranging from 1800 to 1000 kg/m^3 for NPE and SLS foam respectively when optimized concentrations are adopted. Similarly bigger size air voids (D90) varies from 214 to 364 microns and 299 to 554 microns for foam concrete with NPE and SLS surfactants respectively. Hence, the air void size distribution is found to be relatively smaller in foam concrete with NPE when compared to that of SLS for all densities of concrete studied. The improved microstructure of concrete with NPE is reflected in higher strength and lower water permeation characteristics of foam concrete with NPE when compared to that of SLS for most of the mixes studied. Furthermore from the correlation studies it is observed there is significant increase in air void size and reduction in circularity with reduction in density of concrete. Also it is apparent that compressive strength has a direct relationship with density and decreases significantly with reduction in density of concrete and increase in foam volume. Comparative studies on addition of CMC indicates a significant reduction in air void size in concrete which results in 30% enhancement in strength and 45% reduction in sorption characteristics for foam concrete with CMC when compared to mixes without CMC. Examination of influence of density on sorptivity, reveals that sorptivity of foam concrete with SLS reduces significantly with reduction in concrete density from 1800 to 1000 kg/m^3. However NPE foam concrete mixes shows a contradictory trend with higher sorptivity value for lower density concrete.

Chapter 7
CONCLUSIONS AND SCOPE FOR FUTURE WORK

7.1 CONCLUSIONS

The salient conclusions arising out of this research work are summarised in this section. The studies on foam concrete described here basically comes under three stages viz.; (i) Relative Performance evaluation of foam produced with different methodologies and different surfactants (ii) Relative workability behavior of foam concrete with different surfactants (iii) Air void parameters and its influence on compressive strength, water absorption and sorptivity of foam concrete.

The conclusions from the experimental investigations are grouped under these three sections, which are applicable to the characteristics of the materials used and the range of parameters investigated.

7.2 RELATIVE PERFORMANCE EVALUATION OF FOAM PRODUCED WITH DIFFERENT METHODOLOGIES AND DIFFERENT SURFACTANTS

- Studies on relative foam behaviour indicates, that the adoption of higher levels of SLS and CMC concentration are recommended for both methods of production (stirrer and foam generator) to produce highly viscous surfactant solution which can result in stable foam with lesser liquid fraction and smaller bubble size microstructure.
- For the compressed air method of foam production, the IFD and foam drainage reduces significantly with increase in Foam Generation Pressure (FGP), indicating that the effect of FGP is very predominant compared to that of other foam

production parameters. Further the adoption of higher FGP results in foam with smaller size bubble microstructure.

- The foam produced using generator is relatively dry foam and meets ASTM C 796 requirement of initial foam density of 32 to 64 kg/m^3 at the optimum levels of foam production parameters.
- Based on comparative studies on foam production methods, it can be concluded that foam produced based on compressed air method (foam generator) is of better quality with lesser liquid fraction and drainage compared to that of foam produced with stirrer. Also the potential of CMC in improvement of quality of foam (reduction of liquid fraction) is utilized to a greater extent in foam produced using foam generator based on compressed air method. Hence the further studies are carried out with foam generator and upcoming conclusions are applicable only for compressed air method of foam production.
- Comparative studies on surfactants SLS and NPE indicates that increase in CMC concentration results in significant increase in IFD for foam produced with NPE surfactant unlike SLS where decreasing trend is observed. This can be attributed to the higher viscosity of NPE surfactant solution.
- NPE surfactant being non-ionic in nature is identified to be with more compatible with additive CMC. This is more evident from the significant enhancement in viscosity of NPE surfactant solution due to addition of CMC.
- From the optimization studies, the optimal SLS and CMC concentrations are identified as 5% and 0.2% while the optimum NPE and CMC concentrations are 6.1% and 0.16% respectively. However for both the surfactants the optimum FGP is fixed as 490 kPa to get uninterrupted foam production.
- At the established optimal surfactant and CMC concentration, the requirements of ASTM C 869 with respect to foam density, fresh density, compressive strength and water absorption of foam cement paste of density 641 kg/m^3 are fulfilled.

7.3 Relative Workability behavior of Foam Concrete with Different Surfactants

- For the foam concrete produced with both the surfactants, for a constant water-solids ratio, the spread reduces and flow time increases with an increase in foam volume.
- The w-s ratio requirement reduces marginally with increase in density indicating that the workability is influenced by the foam content.
- The spreadability requirement to attain the desired design density ranges from 75 to 150% and 45 to 125% for surfactants NPE and SLS respectively. Similarly for flow time requirement to produce stable mix varies from 4.4 to 7.1 seconds and 4.6 to 9.5 seconds for NPE and SLS respectively. The above spread and flow time values are achieved for foam concrete mixes produced with optimum levels of surfactant and additive concentration.
- Studies on relative workability behaviour of different surfactants indicates that for a constant water solid ratio, the spread of foam concrete made with NPE is 44% higher than that of concrete made with SLS.
- Experimental investigations indicates that the increase in surfactant and CMC concentration which increases the viscosity of surfactant solution also affects the workability related characteristics of concrete. This is evident from the reduction in spread and increase in flow time of foam concrete mixes with increase in surfactant and CMC concentration.

7.4 Air void Parameters and its Influence on Compressive Strength, Water Absorption and Sorption Behaviour of Foam Concrete

- The median air void size (D50) varies from 88 to 126 microns and 97 to 133 microns for foam concrete with fresh density ranging from 1800 to 1000 kg/m^3 for NPE and SLS foam respectively when optimized concentrations are adopted. Similarly bigger size air voids (D90) varies from 214 to 364 microns and 299 to 554 microns for foam concrete with NPE and SLS surfactants respectively. Hence, the air void size distribution is found to be relatively smaller in foam concrete with NPE when compared to that of SLS for all densities of concrete studied.

- The improved microstructure of concrete with NPE is reflected in higher strength and lower water permeation characteristics of foam concrete with NPE when compared to that of SLS for most of the mixes studied.
- Comparative studies on addition of CMC indicates a significant reduction in air void size in concrete which results in 30% enhancement in strength and 45% reduction in sorption characteristics for foam concrete with CMC when compared to mixes without CMC.
- Studies on variation of air void characteristics with density of concrete indicates that there is significant increase in air void size and reduction in circularity with reduction in density of concrete. Also it is apparent that compressive strength has a direct relationship with density and decreases significantly with reduction in density of concrete and increase in foam volume.
- Examination of influence of density on sorptivity, reveals that sorptivity of foam concrete with SLS reduces significantly with reduction in concrete density from 1800 to 1000 kg/m^3. However NPE foam concrete mixes shows a contradictory trend with higher sorptivity value for lower density concrete.

7.5 Scope for Future Work

The area on which continued research can be undertaken to provide better understanding of the material and thus be of more use to the construction industry are:

i) In order to integrate foam concrete into residential concrete, studies can be made on properties of light weight blocks, wall panels and lightweight reinforced foam concrete.

ii) Studies on identification of more compatible chemical admixtures in foam concrete to reduce the water-solids ratio requirement can be carried out.

iii) Studies on microstructure of concrete (chemical analysis) in order to analyze the impact of addition of CMC and relative performance of SLS and NPE in concrete can be carried out.

iv) Investigations on use of mineral admixtures such as fly ash and slag can be made to improve the mechanical and durability related performance of foam concrete.

v) Investigations on functional characteristics of foam concrete namely thermal conductivity and sound transmission can be undertaken.

vi) Methods of reducing shrinkage of foam concrete by incorporation of light weight coarse aggregates and shrinkage reducing admixtures can be investigated.

vii) Full scale long term durability test to study the behaviour of foam concrete in sulphate and chloride environment can be conducted.

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PUBLICATIONS/ AWARDS/ POSTER BASED ON RESEARCH

1. Sahu, S. S., Gandhi, I. S. R., and Khwairakpam, S. (2018). "State-of-the-Art Review on the Characteristics of Surfactants and Foam from Foam Concrete Perspective." *Journal of The Institution of Engineers (India): Series A*, 99 (2), 391-405.
2. Received Best Poster Award "Studies on assessment of suitability of commonly available surfactants for foam concrete production" in Research Conclave, 8-11 March (2018), organized by Students' Academic Board, IIT Guwahati.
3. Sahu, S.S., Gandhi, I. S.R. "Evaluation of performance of foam produced with different methodologies for use in foamed concrete production" 4th International Conference on Civil Engineering and Materials Science (ICCEMS 2019) held in Bangkok, Thailand during 17th to 19th May, 2019.
4. Sahu, S.S., Gandhi, I. S. R. "Performance evaluation of foam produced with surfactant Sodium lauryl sulphate and additive Carboxy methyl cellulose for use in foamed concrete." *Journal of The Institution of Engineers (India): Series A* (*Under Review*)
5. Khwairakpam, S., Gandhi, I. S. R., and Sahu, S.S. "A state of the art review on shrinkage behaviour of foam concrete."*Journal of Structural Engineering*. (*Accepted*)
6. Sahu, S. S., Khwairakpam, S., Gandhi, I. S. R. "Influence of properties of surfactants and foam on foam concrete behaviour- A critical review." in Research Scholar Symposium - Concrete Research in India (CRI) on 13th December (2018), organized by IIT Bombay, Mumbai.
7. Sahu, S.S., Gandhi, I. S. R. "Studies on influence of characteristics of surfactant and foam on foam concrete behaviour." *Construction and Building Materials* (Manuscript under preparation)