NOVEL INVESTIGATIONS ON POTENTIAL OF BALANITES AEGYPTIACA AND SESAMUM INDICUM FOR SURFACTANT PREPARATION AND APPLICATION IN FOAM CONCRETE

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KHWAIRAKPAM SELIJA

DEPARTMENT OF CIVIL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY GUWAHATI

GUWAHATI-781039, INDIA

March, 2023

Dedication

To my

Family and Well-wishers

Indian Institute of Technology Guwahati

Department of Civil Engineering

Statement

I, hereby declare that the content embodied in this thesis entitled ***"Novel Investigations on Potential of Balanites Aegyptiaca and Sesamum Indicum for Surfactant Preparation and Application in Foam Concrete"*** is the result of investigations carried out by me at the Department of Civil Engineering, Indian Institute of Technology Guwahati, Guwahati, India.

In keeping with the general practice of reporting scientific observations, due acknowledgements have been made wherever the work described is based on the findings of other investigators.

Date

Khwairakpam Selija

Place: IIT Guwahati

Indian Institute of Technology Guwahati

Department of Civil Engineering

Certificate

This is to certify that the thesis entitled ***"Novel Investigations on Potential of Balanites Aegyptiaca and Sesamum Indicum for Surfactant Preparation and Application in Foam Concrete"*** *by Khwairakpam Selija (Roll No: 156104022), to the Indian Institute of Technology Guwahati, India, for the award of Doctor of Philosophy, is a record of bonafide research work carried out by her under my guidance and supervision. The work embodied in this thesis has not been submitted for any other degree or diploma. In my opinion, the thesis is up to the standard of fulfilling the requirements of the doctoral degree as prescribed by the regulations of this Institute.*

Date:

Place: IIT Guwahati

Dr. G. Indu Siva Ranjani

Assistant Professor

Department of Civil Engineering

Indian Institute of Technology Guwahati

Guwahati-781039, Assam, India

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Khwairakpam Selija

Abstract

Synthetic surfactants commonly used in foam concrete production is reported to exhibit serious environmental threat. Hence utilization of natural surfactants could present new sight in improving the environmental impact of foam concrete. However, studies on valorization and extraction of natural surfactants from different plant and animal sources for use in foam concrete is very scarce from Indian context. Considering the above facts, the current study aims to use natural surfactants valorized and extracted from two different biological sources, namely sesame seed (rich in protein) and hingot fruit (rich in saponin) as prospective supplementary alternative to conventional synthetic foaming agents in foam concrete production. Balanites aegyptiaca also known as hingot is found all over the world and studies on phytochemistry of various parts of plant indicate that fruit, seeds and bark part of plant has 22-27% of saponin. Adding to above, excellent plant's adaptability, high yield features and abundant availability are other added reasons for selection of this plant for this study as it can add economical as well as environmental benefits. Another plant source used for surfactant extraction in the present study is Sesamum indicum commonly known as sesame seed. Studies have proved that sesame seed has rich protein content of 20 to 25% and hence has excellent potential foamability. Despite the huge potential foamability of above surfactants (sesame and hingot), it is surprising to find that to date, its potential has not been assessed for use in foam concrete production.

The first phase of present study comprises of the optimization of surfactant extraction and foam production processes through systematic experiment design based on response surface methodology (RSM). The parameters considered concerning the extraction process of sesame and hingot surfactants and foam production are surfactant concentration, temperature and duration of heating and FGP. However, for sesame surfactant extraction process, adding to above, sodium hydroxide concentration (alkali for hydrolysis) is also considered as additional parameter. The possible response surface models have been developed for prediction of various foam and surfactant characteristics such as initial foam density (IFD), foam stability, foam bubble size and viscosity of surfactant solution. The validity of models is tested by ANOVA and verified experimentally. Analysis of influence of various input parameters on responses indicate that surfactant concentration has significant effect on all the responses studied. Further the parameters associated with heating process (heating temperature and duration) also have substantial influence on various foam and surfactant characteristics as it promotes the hydrolysis of protein and saponin. The obtained results established that increase in surfactant concentration, heating temperature and heating duration of surfactant solution resulted in increase in viscosity of surfactant solution eventually leading to reduction in foam liquid fraction, foam drainage and bubble size. Hence good correlation is established between above mentioned foam and surfactant parameters. General comparison between two surfactants shows that sesame surfactant is highly viscous when compared to hingot. Further, for compressed air foam, higher level of foam generation pressure is needed for thorough mixing of air and liquid which eventually results in good quality foam with reduced liquid fraction, slower drainage rate and smaller size foam bubble microstructure. Hence the above results indicate that optimization of various input parameters of surfactant extraction and foam production processes are needed to improvise the quality of foam. Multiple optimization approach is adopted using Design Expert with different constraints set for various parameters. RSM approach predicted the optimum combination of input parameters for sesame surfactant as 6% surfactant concentration, 490 kPa FGP, 6% NaOH concentration, 90^{o}C heating temperature and 5hrs heating duration. Similarly, for hingot surfactant the predicted parameters are 6% surfactant concentration, 550 kPa FGP, 90^{o}C heating temperature and 5hrs heating duration. Furthermore, performance of surfactants in foam cement paste is assessed as per ASTM requirements and it is observed that both the surfactants are suitable for use in foamed concrete production when optimized foam production parameters are adopted.

Another main facet of this work is to carry out an extensive experimental investigation on the effect of water-solids ratio on the various characteristics of foam concrete on which only very limited studies are available in literature. The role of water-solids ratio on various physical, chemical and mechanical properties of foam concrete have been considered for range of densities. The results show that performance of foam concrete with different foaming agents varies greatly due to creation of different void structure in concrete. Comparative studies on consistency of foam concrete with two different surfactants indicates that the spread and flow time of hingot foam concrete mixes are relatively higher and lower respectively than that of sesame foam concrete mix for most of the mixes studied. The enormous difference observed here can be attributed to relatively lesser viscous nature of hingot surfactant solution when compared to sesame surfactant. Further consistency of foam concrete is found to increase with increase in foam volume contrary to some of the earlier studies reported in the literature.

The air void characterization of foam concrete is done based on air void volume, shape and size. Experimental studies on air void characterization reveal that a significant increase of 35 % in D90 (bigger size air voids) is observed with an increase in water-solids from 0.25 to 0.32 for sesame foam concrete with target density 1000 kg/m3. In addition, closer examination of pore size distribution highlights that in comparison to sesame surfactant, hingot surfactant foam concrete mixes exhibit bigger size air voids, which can be ascribed to lesser viscosity of hingot surfactant solution. In line with above observations, sesame surfactant solution which is relatively more viscous than hingot shows higher circularity air voids which is particularly noticeable in mixtures with higher foam volume. Another important observation is that within the range of water-solids ratio which results in stable mixes, it is evident that for most of the mixes studied, the compressive strength of foam concrete increases with water-solids ratio first and then decreases. Hence, there is an optimum water-solids for a particular density of foam concrete which results in maximum strength. The mechanical behaviour of foam concrete is found to depend on both porosity and pore size distribution. Hence, the optimized water-solids ratio derived for various mixes is justified with air void structure. In line with earlier findings, mixes with optimum water-solids are also found to exhibit minimum water absorption. The compressive strength and water absorption of sesame foam concrete with target density 1800 kg/m^3 is 56 % higher and 7 % lower respectively than that of hingot foam concrete. The above results corroborate the air void characterization results, which proved bigger size air voids in hingot foam concrete. Further, for a greater insight of concrete microstructure, XRD and EDS are employed to study the mineral phases of concrete. The results of mineralogical analysis proved that mixes with higher strength showed higher peaks of Portlandite and lower Ca/Si ratio indicating greater amount of hydration.

Keywords: Foam concrete, natural surfactant, hingot fruit, sesame seed, density, density ratio, surfactant concentration, foam generation pressure, foam stability, viscosity of surfactant solution, response surface methodology, water solids ratio, air void parameter, image analysis, compressive strength, water absorption, mineralogical

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Notations

AMCSD	American Mineralogist Crystal Structure Database
BM	Base mix
$Ca(OH)_2$	Portlandite
CCD	Central composite design
CMC	Critical micellar concentration
C-S-H	Calcium Silicate Hydrate
CTAB	Cetyltrimethylammonium bromide
C50	Median circularity
C10	Irregular air void
DD	Dry density
DSC	Differential scanning calorimetry
D50	Median air void
D90	Bigger size air void
EDS	Energy Dispersive X-ray spectroscopy
FC	Foam concrete
FER	Foam expansion ratio
FGP	Foam generation pressure
FS	Foam stability
FTIR	Fourier Transform Infrared spectroscopy
GGBS	Ground granulated blast furnace slag
GSAS	Generalized Structure Analysis System
HM	Hingot mix
i	Cumulative volume absorbed per unit area of inflow surface
LWAC	Light weight aggregate concrete
LWC	Light weight concrete
NaOH	Sodium hydroxide
NH_4Cl	Ammonium chloride
NPE	Nonyl phenyl ether
PP	Polypropylene fiber
PVA	Polyvinyl alcohol
RSM	Response surface methodology

R^2_{adj}	Adjusted R squared
SEM	Scanning electron microscopy
SF	Silica fume
SLS	Sodium Lauryl Sulphate
SLES	Sodium lauryl ether sulfate
SM	Sesame mix
SP	Super plasticizer
TGA	Thermogravimetric analysis
IFD	Initial foam density
IFGRI	International Plant Genetic Resources Institute
w/c	Water-cement ratio
w/s	Water-solids ratio
WRA	Water reducing admixture
XRD	X-ray diffraction
XRT	X-ray tomography

Chapter 1

Introduction

1.1 General

In the modern era, building materials are increasingly directed towards the lightweight and best able to meet structural design and constructional productivity. For this purpose, lightweight concrete has been developed in several forms possessing various combinations of structural strength, shock mitigation, weight reduction, heat insulation, fire resistance and ease of handling. Therefore, it is not surprising that versatile materials with these possibilities, when satisfactorily combined for economy or performance, are steadily gaining acceptance in regional building projects. Although reinforced autoclaved aerated concrete panels are readily available, their application is limited because of the heavy investment associated with the autoclaving process. Hence, as the output of the search for alternative materials which can be both economical and environmentally sustainable, research groups have developed foam concrete a few decades back.

Foam concrete, a lightweight concrete, is a cementitious material with a minimum of 20 % volume of mechanically entrained foam (Barnes, 2008). Although foam and conventional concrete are mostly similar, the main difference is that the coarser aggregates in the former are eliminated. Based on the method adopted for production, foam concrete is classified as pre-foamed and mixed foam. The pre-foamed method involves the use of a foam generator with which stable foam is produced, after that, the foam is added to the base mix and mixed thoroughly to produce the homogeneous cellular structure of concrete (Nambiar and Ramamurthy, 2007). In contrast, in the mix foam method, the cellular structure is produced by rapid mixing of the foaming agents along with other base mix ingredients. As the cement grains on the surface of the air bubble hydrate, a porous cellular structure is formed. This porous structure contributes to all positive attributes such as lightweight and higher insulating nature compared to normal-weight concrete. Another advantage of foam concrete is that it can be tailor made to meet different applications with a wide range of densities (300-1850 kg/m^3). The unique properties of this cellular concrete have paved the way for various applications such as sound barriers and firewalls, foundations, building panels, and filling applications such as structural backfill, geotechnical fill and mine fill. Adding to the above, there are also evidences of applications related to its energy absorption capacities, such as vehicle arresters on airport aprons, mine plugs, ballistic range targets, and roadway crash barriers.

1.2 Background of Foam Concrete

If the history of foam concrete is looked into, literature shows that the usage of natural foaming agents can be dated back to Roman times, and there is evidence that Romans used animal blood as air entrainers. The Egyptians also used this type of technology over 5000 years ago (Aldridge, 2005). Although the material was first patented in 1923 (Brady et al., 2001), widespread use of the material has been reported only over the past 20 years. Advancements in technology in terms of production equipment and better-quality surfactants have aided the use of foam concrete on a larger scale. Though it is reported to be extensively used in foreign countries like Netherland, Sweden, Germany, Switzerland, the USA, and the UK (Cox and Van Dijk, 2002), its usage in India is limited. When the history of foam concrete in India is considered, the contribution made by Singh is noteworthy. In India, Singh attempted a successful trial on use of foam concrete in a G+3 storey building in Delhi (Singh, 2005).

Recently attempts have been made to extend the application of foam concrete for structural purposes. In this context, researchers have tried various techniques to improve the strength of foam concrete. Research output has established that uniform cellular structure is a key factor in producing concrete with good mechanical properties. In this direction, researchers (Abd Elarahman et al., 2021; Wang et al., 2020; Siva et al., 2017; Hilal et al., 2015b) improved the cellular structure of concrete through the addition of additives which prevents early collapse of bubbles. In this regard, recent studies have proved that it is possible to achieve reasonably acceptable strength performance through modification of cementitious matrix by incorporating fine particles such as silica fume, fly ash and polypropylene fibres (Gowri and Anand, 2018; Hilal et al., 2015b; Bing et al., 2012).

Figure 1-1 shows the (cumulated) number of empirical, peer-reviewed papers published on foam concrete from 1958 to 2021. From Figure1-1, it is evident that the academic interest in foam concrete is steadily increasing. Further, it is to be noted that the scientific output of foam concrete-related papers has increased significantly over the last ten years.

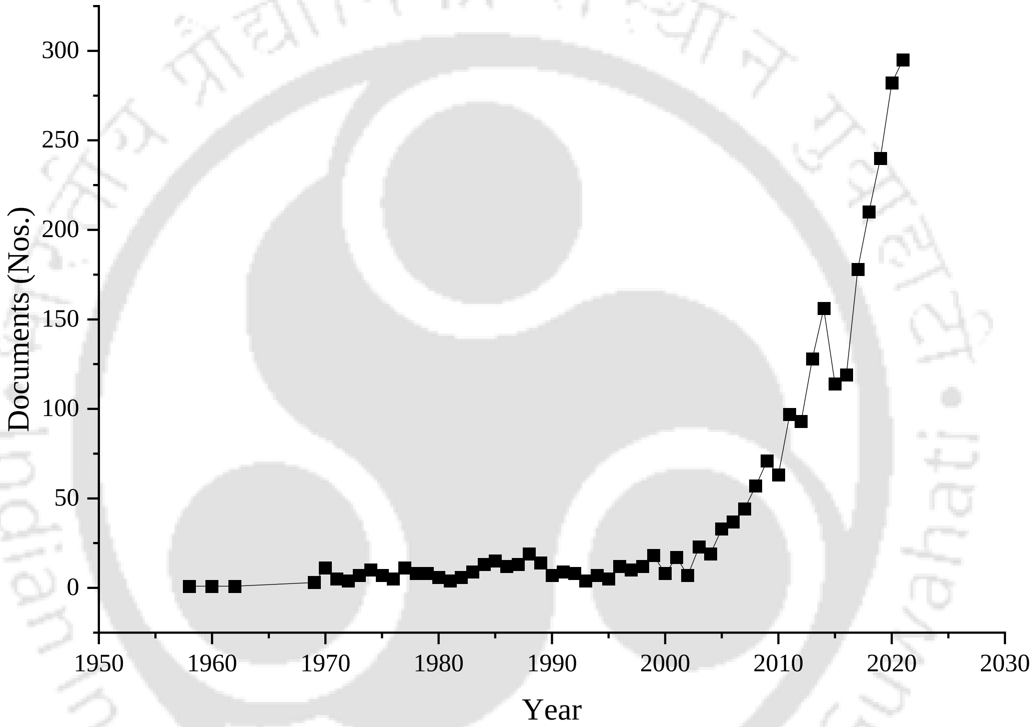


Figure 1-1 Peer-reviewed documents published on foam concrete

1.3 Advantages

The foremost advantage of foam concrete is the compatibility of material for both cast in situ and precast applications. Also, many industrial wastes like fly ash and slag can be consumed in foam concrete production without much effect on its mechanical properties, making it more environmentally friendly (Kearsley and Wainwright, 2001a; Wei et al., 2013). Considering the density of foam concrete is much lower than that of conventional concrete, a structure constructed with foam concrete would undoubtedly be significantly lighter. This leads to tremendous savings in reinforcement steel in the foundations and structural members and additional savings in transportation and handling activities, and raw materials, as no coarse aggregate is required to produce foam concrete. Another main contribution of foam concrete, particularly in harsh climate conditions, is its thermal insulation, which helps to achieve energy efficiency in buildings. The cellular structure of foam concrete (porosity, pore size distribution, and pore structure) significantly impacts its thermal conductivity (Kudyakov and Steshenko, 2014). Also, thermal conductivity is less for concrete with smaller pores than concrete with larger pores due to the reduction in heat flow due to convection. Even a small decrease in dry density by 100 kg/m^3 is reported to reduce thermal conductivity significantly (Huang et al., 2015; Pan et al., 2014; Weigler and Karl, 1980a).

1.4 Applications

Foam concrete has found applications in many civil engineering areas due to its distinctive properties such as density reduction, high thermal insulation, self-compacting nature and ease of production (Just and Middendorf, 2009; Ramamurthy et al., 2009). Mostly low density foam concrete finds its use in insulation and filling applications, while high density foam concrete has been used in structural applications (Just and Middendorf, 2009; Ramamurthy et al., 2009; Tikalsky et al., 2004). Its excellent flowing nature has made foam concrete material of choice for abundant fills such as old sewers, storage tanks, basements, ducts, and voids under roadways (Fu et al., 2020; Mindess, 2019; Amran et al., 2015). Other commonly reported sectors of use are lightweight blocks and precast panels, fire insulation, thermal and acoustic insulation, road sub-base, trench reinstatement, soil stabilization, and shock-absorbing barriers for airports and regular traffic (Just and Middendorf, 2009; Ramamurthy et al., 2009; Uddin et al., 2006). Table 1-1 categorises the foam concrete applications based on its density.

Table 1-1 Summary of application of foam concrete based on its density (Sari and Sani, 2017)

Density (kg/m^3)	Application
300-600	Soil stabilization, raft foundation.
600-800	Widely used for filling applications such as old sewerage pipes, wells, basements and subways and as an alternative to granular fill.
800-900	Principally used in the manufacture of blocks and other non-load bearing elements of the building such as balcony railings, partitions, and parapets.
1100-1400	Used for cast-in-place construction and the prefabrication of walls, floor screeds, and load-bearing or non-load-bearing walls.
1500-1600	Suitable for slabs and other load-bearing building elements that require a higher level of strength.

1.5 Outline of the Thesis

The thesis is divided into five chapters. The first chapter (Chapter 1) provides a brief overview of foam concrete, its benefits, and its applications. The second chapter (Chapter 2) provides a critical review of surfactant and foam characteristics in foam concrete production. Furthermore, a review of various foam concrete production methods, foam concrete properties, and major research gaps where systematic research is needed is also highlighted. The objective and scope of the current study are derived from the critical review and presented in the third chapter (Chapter 3).

Through a systematic experimental design based on response surface methodology (RSM) the fourth chapter (Chapter 4) demonstrates the optimization of extraction processes of natural surfactants from sesame seed (*Sesame indicum*) and hingot fruit (*Balanites aegyptiaca*). Various aspects of surfactant and foam are discussed in-depth, including initial foam density, foam stability, foam bubble size, and surfactant solution viscosity. The fifth chapter (Chapter 5) depicts a more in-depth experimental programme on the comparative analysis of the properties of foam concrete made with the two natural surfactants mentioned above. The first section of this chapter examines the effect of the water-solids ratio on the fresh state properties of foam concrete for mixes with varying foam volumes and two different natural surfactants. The effect of varying the water-solids ratio on the pore structure and the resulting impact on the mechanical behaviour of concrete and water permeation characteristics is then investigated. For various densities of foam concrete, the optimum water-solids ratio for satisfactory fresh concrete characteristics, mechanical properties, and water permeation characteristics is determined. Further research on the microstructure is used to support the experimental results. The last chapter (Chapter 6) presents the typical conclusions drawn from the studies and the scope for future research.

Chapter 2
Literature Review

2.1 General

With global warming on the rise, the construction industry is looking for an alternative to ordinary concrete due to its high dead weight and thermal conductivity. Research is being conducted in various directions, and the current emerging trend is the use of foam concrete, a lightweight cellular concrete. Foam concrete is fundamentally a mortar or cement paste with air voids artificially entrapped in it. Commonly, foam concrete is well known for its special characteristics such as low self-weight, high flowability, and exceptional thermal insulation properties. Because of the simpler mechanism of air incorporation, foam concrete is reported to be the most cost-effective and controllable pore-forming process. The pores are introduced mechanically either through preformed foaming or mix foaming methods. Detergents, resin soap, glue resins, saponin, and hydrolyzed proteins such as keratin, are commonly used foaming agents in concrete (Narayanan and Ramamurthy, 2000).

There are various review articles published on the history of foam concrete, the constituent materials used, foam concrete properties, construction applications, and important projects completed worldwide (Amran et al., 2015; Ramamurthy et al., 2009; Jones and McCarthy, 2005c). Though these reviews cover important functional properties such as fire resistance, thermal conductivity, and acoustic properties, literature on fresh state properties, durability, and the air-void system of foam concrete is very scarce.

2.2 Review on surfactant

Surfactants are molecules that have both hydrophilic (water-loving) and hydrophobic (water-hating or oil-loving) components. The above structure of surfactants allows them to spontaneously self-assemble in a variety of solutions or augment the interface to reduce interface tension (Ghosh, 2009). Surfactants have a wide range of applications and they are used as essential additives in producing materials that are commonly used in daily life such as laundry detergents, industrial cleaners, and cosmetics. Some surfactants find their use in the pharmaceutical, textile, food, and petroleum industries as wetting, emulsifying, defoaming, or solubilizing agents (Figure 2-1). In this line, it is also used in the production of stable foam for use in foam concrete. All surfactants do not produce a stable foam that can be used to make foam concrete. As a result, surfactant selection is critical concerning the properties of foam such as foam density, foam output rate, foam capacity, and foam stability, all of which eventually influence foam concrete properties. Various surfactant properties are discussed in detail in the subsequent sections, along with their classification and influence of various parameters.

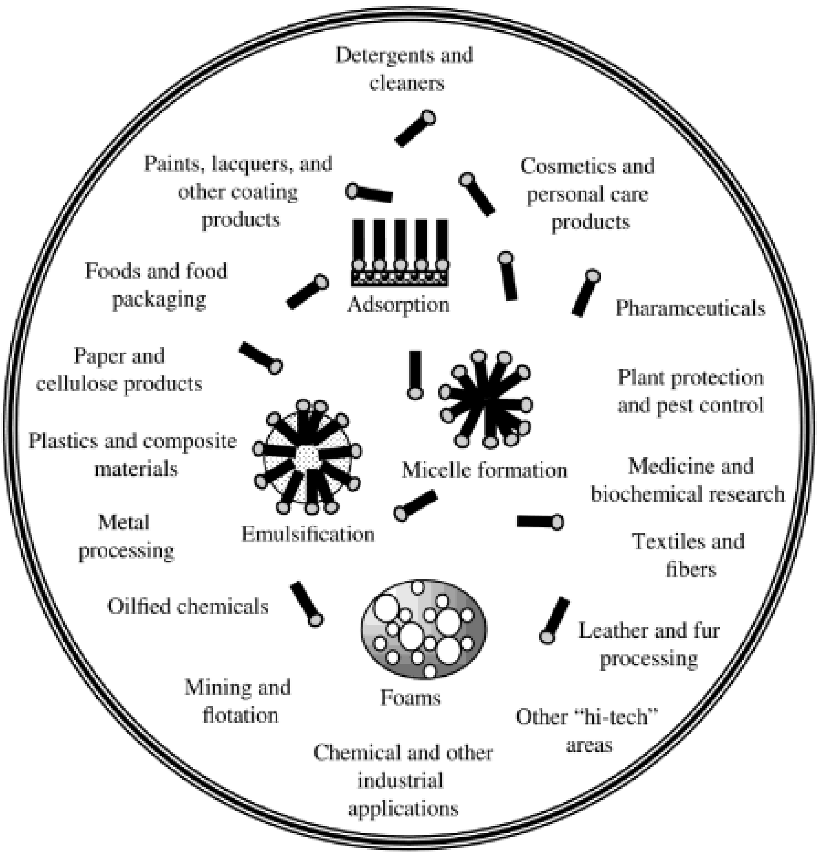


Figure 2-1 Some important, high-impact areas of surfactant applications (Myers, 2020)

2.2.1 Surfactant properties

Surfactant properties significantly impact foam properties because they affect surface tension and gas-liquid interfacial properties. The following paragraphs discuss important surfactant properties, such as adsorption, solubility, and viscosity.

Critical micellar concentration (CMC) and the effect of various factors on CMC

The surface-active effects of foaming are caused by the adsorption of surfactants at the air/liquid interface. Further, the concentration of a surfactant in a solution affects its adsorption onto a surface. The CMC of a surfactant is a significant parameter because it defines the concentration at which monolayer adsorption is complete and surface-active properties are optimal. When the concentration exceeds CMC, the molecules no longer have any available adsorption site and thus form micelles (as shown in Figure 2-2) which have no surface activity (Gecol, 2006). As a result, there is a lot of practical interest in the CMC because it represents the minimum concentration required to get the maximum benefit in terms of surface activity. The CMC is determined by the chemical structure of surfactant and is said to decrease as the number of carbons in a homologous series chain increases (Liu et al., 2008). Hence the practical formulae should be designed by moderating surface-active properties and solubility. There are also various other factors reported to affect CMC such as pH, temperature etc. An increase in pH will increase CMC while an increase in temperature is reported to reduce CMC.

Viscosity

Surfactant concentration has a significant influence on its viscosity and this can be attributed to the variation in micellar structure. At lower concentrations, the presence of spherical micelles results in low viscosity. However, with an increase in surfactant concentration, micelles change shape to rods, cylinders and even hexagonal packing which results in a significant increase in viscosity. On a similar note, the addition of electrolytes can also result in a change of spherical micelles into cylindrical micelles resulting in a spectacular increase in viscosity. It has also been discovered that the chain length of surfactants influences the viscosity of the solution. Straight-chain alkyl surfactants have a higher viscosity than branched-chain surfactants, with a smaller surface area (Rosen and Kunjappu, 2012). Researchers have also demonstrated that foam made with a more viscous surfactant solution has a higher initial density and is more stable over time. According to reports, the viscosity of the solution is proportional to the size of the bubbles. The features of foam concrete produced with the high viscous foaming solution in both the fresh and hardened states were found to be desirable, illustrating the efficacy of increasing foaming solution viscosity.

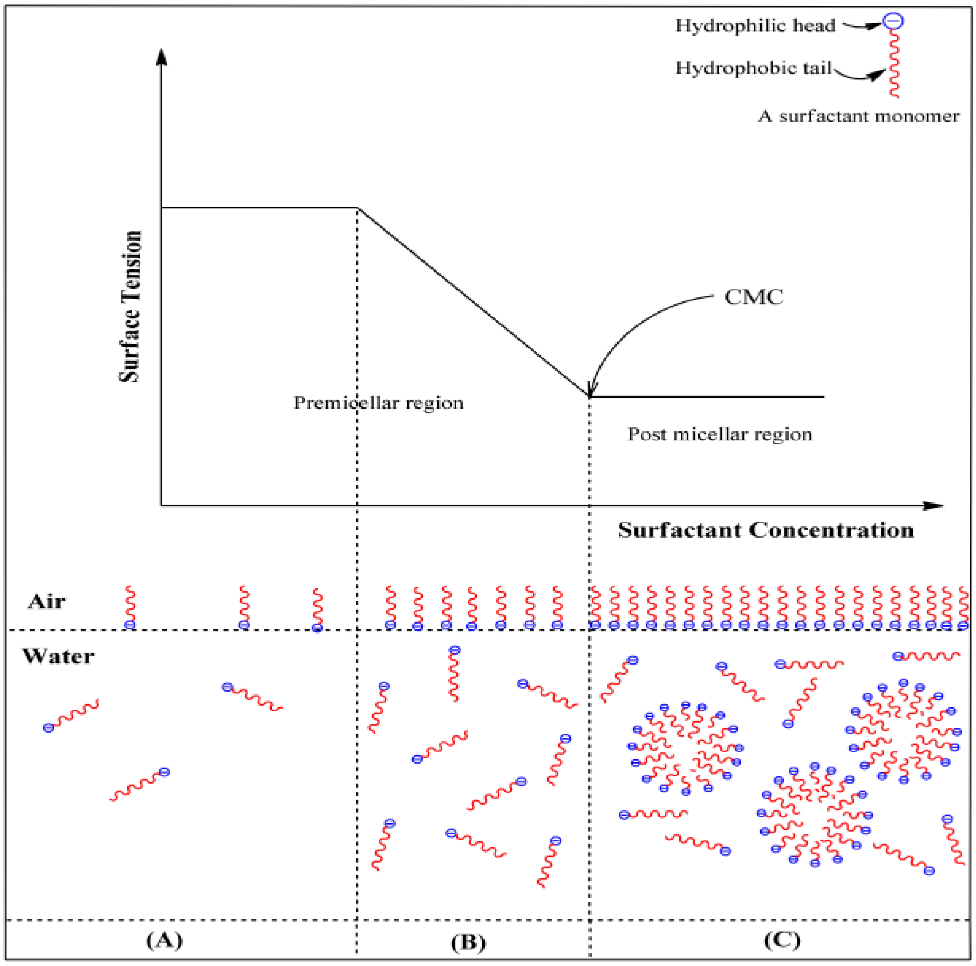


Figure 2-2 Surfactant activity at different concentrations (Rai et al., 2021)

2.2.2 Surfactant classification

There are several ways in which surfactants can be classified as shown in Figure 2-3. The simplest method for classifying surfactants is based on their origin.

Synthetic vs Natural surfactants

Based on their origin, surfactants can be divided into natural and synthetic types. Generally, synthetic surfactants are used in a variety of industries such as textiles, agriculture, cosmetics, food, leather, paper, paint, pharmaceuticals, and petrochemicals/ petroleum. The greater degree of application of synthetic-type foaming agents can be attributed to their high productivity in the manufacturing process, strict control over their composition, and longer storage life (Ranjani and Ramamurthy, 2010a). Sodium Lauryl Sulphate (SLS) (Ranjani and Ramamurthy, 2010a), Cetyltrimethylammonium bromide (CTAB) (Samson et al., 2017), Sulfanol (Ranjani and Ramamurthy, 2010b), and Sodium oleate are some of the synthetic type foaming agents commonly used in foam concrete production (Corr et al., 2002). One of the disadvantages of synthetic surfactants as reported in the literature is their incompatibility with other admixtures. Foam concrete's self-flowing characteristic needs the application of a water-reducing additive (WRA) to reduce water demand. The dosage of WRA in concrete, on the other hand, needs to be carefully researched because it affects the stability of foam made using synthetic surfactants (Jones and McCarthy, 2006). Further, the process of manufacturing synthetic surfactants involves harsh conditions such as high pressure (hydrogenation for the fatty alcohol process), nitrogen protection (ethoxylation), and blended catalysts (alkylation, oxidation, ethoxylation, condensation, and polymerization) which is harmful to the environment. Also, synthetic surfactants prepared from petrochemicals frequently result in byproducts or isomers of the main products (e.g., ortho- or meta- in linear alkyl sulfonates, disulfonates in petrosulfonates or methyl ester sulfonates), causing environmental, or ecological problems (Nnanna et al., 2001). Since 1970, synthetic surfactants have been known to pose chronic and sub-lethal toxic effects on aquatic animals (Lewis, 1991) and health hazards to people (Zhou et al., 2013).

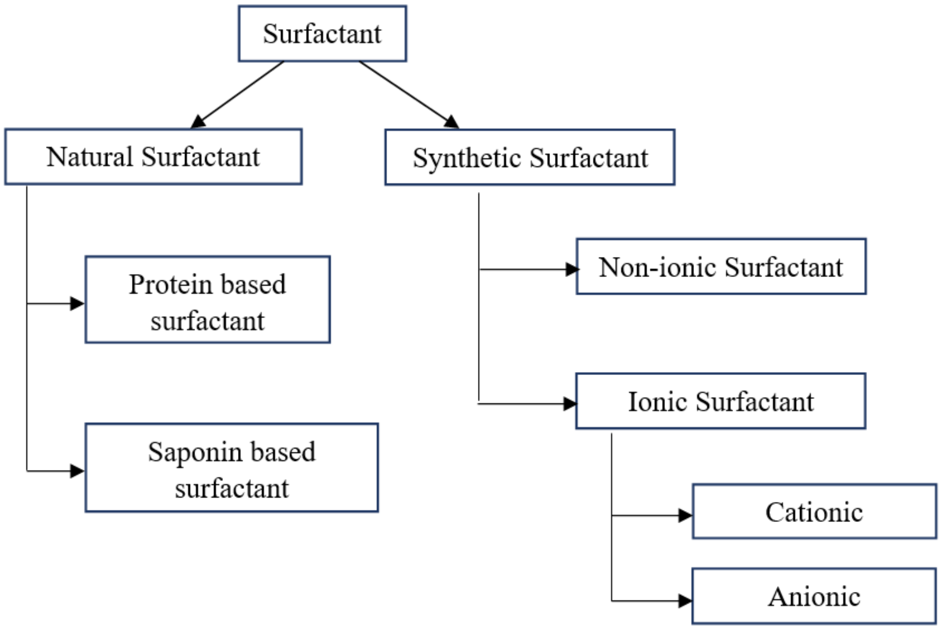


Figure 2-3 Classification of surfactants

The above-mentioned disadvantages of synthetic surfactants have promoted the use of natural renewable surfactants. Natural surfactants are biological compounds derived from various waste sources, including animal and vegetable waste. For instance, there are studies reported on the extraction of natural surfactants from various sources such as Glycyrrhiza Glabra (Arabloo et al., 2015) and Zyziphus Spina Christi (Shahri et al., 2012) for use in the oil recovery, Sapindus Mukorossi (Siva et al., 2017) and Phytolacea Dodecondra (Retta, 1991) for use in foam concrete. Many plant products are naturally surface-active because of a compound known as "saponin,". Saponin is present in various parts of many plant species. Soaproot (Chlorogalum pomeidianum), soapbark (Quillayasaponaria), soapberry (Sapindus saponaria), soapnut (Sapindus mukurossi), and soapjacob (Glinus lotoides) are some of the saponin-rich plant species (Siva et al., 2017; Oleszek and Hamed, 2010). Saponins are also reported to be found in the grains of various important pulses and legumes, including soya beans, chickpeas, mungbeans, peanuts, lentils, and beans (Singh et al., 2017). Other edible plant species rich in saponin include oats, leeks, garlic, asparagus, tea, spinach, sugar beet, sesame, and yam (Oleszek and Hamed, 2010). Table 2-1 shows the list of various plant sources rich in saponin. The combination of the nonpolar sapogenin and the water-soluble side chain causes saponin to foam, which is similar to the structure of most synthetic surfactants with lipophilic and hydrophilic molecular parts.

On the other hand, there are also various plant and animal sources which are rich in protein with potential foamability. Proteins are amphipathic or amphiphilic molecules by nature, which means they have both a hydrophobic (nonpolar) and a hydrophilic (polar) moiety. However, natural proteins are not used as commercial surfactants in their basic form. Rather, proteins are chemically or enzymatically modified to produce products with surface-active properties. Keratin, hoof, horn meal from cattle, fish scales, casein, blood, bones, and other carcass residues are examples of animal protein sources. Various types of plant proteins are available commercially today, including those derived from pulses, cereals and pseudo cereals, oilseeds, tubers and tree nuts, which differ in their level of purity (concentrates or isolates), physical state (native, denatured or hydrolyzed), functionality, nutritional value, allergenicity, available volumes and price (Amagliani et al., 2021; Sá et al., 2020).

The process of manufacturing natural surfactants is not that simple and involves many stages such as pre-treatment, hydrolysis, neutralization, treatment with preservatives, 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, 2008). 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 hydrolysis, the most commonly adopted method is prolonged boiling in a strong acid (hydrochloric acid or sulphuric acid) (Pasupuleti and Braun, 2008) or strong base (sodium hydroxide, lime or calcium hydroxide) (Zhang et al., 2014; Lin et al., 2013) 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 needs 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 the growth of microorganisms. Because of the possibility of degradation by bacteria and other microbes (Brady et al., 2001), the natural source of protein-based foaming agents has to be disinfected with preservatives 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 to obtain a homogeneous (Dickinson, 1997). 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 as shown in Figure 2-4. Despite the complicated manufacturing process there is increasing interest in natural surfactants because of some unique properties like low toxicity and biodegradable nature. In addition to this, it has also achieved fame in the greenhouse effect by reducing CO_2 emissions during its manufacture (De et al., 2015). Also, its performance when used as a 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 the lamellar layer resulting in more stable foam (Brady et al., 2001; Panesar, 2013). The above resulted in enhancement of compressive strength of foam concrete with natural surfactants (Kim and Jeong, 2012; Jones, 2001). Besides these advantages, the natural surfactant also has a 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. Table 2-2 presents an overview of various proprietary surfactants available in the market.

Table 2-1 Saponin source from various plants (Kjellin and Johansson, 2010)

Plant source	**Common name**	**Use**	**Plant parts**	**Concentration (%)**
Aesculus hippocastanum	Horse chestnut	Aescin source	Seeds	10
Agave sisalana	Sisal, henequen, hemp plant	Hecoginin source	Leaves	>12
Balanites aegyptiaca	Heglig, lalob, desert date, hingot	Diosgenin and	Fruits, seeds,	22–27

Plant source	**Common name**	**Use**	**Plant parts**	**Concentration (%)**
		yamogenin source	bark	
Chlorogalum pomeridianum	Soaproot	Amolonin source	Bulbs	19–22
Dioscorea composite	Yams, barbasco	Diosgenin source	Rhizomes, roots	4–6
Glinus lotoides	Soap Jacob, lotus Sweet juice	Hopane and oleanane source	Roots, leaves, seeds	16.5
Gypsophilla paniculata	Baby's breath	Gypsogenin source	Roots	>10
Quillaya saponaria	Quaillaya, Murillo's-bark	Soaps, foaming agents	Bark	≥25
Sapindus mukurossi	Soapnut	Hedragenin source	Fruits and roots	20
Sapindus saponaria	Soapberry	Hedragenin source	Fruits	11
Tribulus terrestris	Puncturevine, yellow vine	Protodioscin source	Fruits	>20
Trigonella faenum graecum	Fenugreek	Diosgenin source	Seeds and leaves	8–10
Yucca schidigera	Mohave yucca, Joshua tree	Soaps, foaming agents	Stalk and roots	10

Table 2-2 Proprietary foaming agents

Proprietary foaming agents	**Country**
Neopor	Germany
Green Froth	Russia
ISOCEM	Italy
FOAMTECH	South Africa
VEPRO 95	France
FOAMSEM	Italy
SDO (PN Brand)	Russia
Lithofoam	Germany
Norait PA-1	Malaysia
FOAM CONCRETE – LITE Foaming Agent	India
Adage (plant based)	India
Green Stone Foaming Agent	India

Ionic surfactants vs non-ionic surfactants

If the surface-active part of molecules carries charges, it is called an ionic surfactant while a surfactant without charge is called non-ionic. Ionic surfactants are further categorised into anionic and cationic surfactants which have negatively and positively charged head groups respectively. Generally, anionic surfactants are used on a greater scale than all other types. Carboxylate soaps are one of the most common forms of anionic surfactants in the market. Soaps are the alkali metal salts of fatty acid carboxylic acids in general. Sodium lauryl sulphate (Narayanan and Ramamurthy, 2012), sodium oleate (Corr et al., 2002), alpha olefin sulphonates (Kim and Jeong, 2012), and sulphanol are some of the carboxylate soaps typically utilised in foam concrete manufacture (Ranjani and Ramamurthy, 2010b). Sulphates are the next most common anionic surfactant class. Fatty alcohol sulphates (e.g., SLS), ether sulphates (e.g., sodium lauryl ether sulfate (SLES)), Nonyl phenyl ether (NPE) and fatty acid alkanoamide ether sulphates are commercial sulphates used as surfactants in foam concrete manufacturing. Ether sulphates are said to perform better than alcohol sulphates, and this is due to the presence of polyethylene oxide, which gives ether sulphates superior solubility (Porter, 1994).

Comparative analysis of ionic and non-ionic surfactants indicates that ionic perform better than non-ionic surfactants. The electrical double layer existing at the interface in ionic surfactants interacts with the opposing interface to create disjoining pressure and eventually results in more stable foam (Myers, 2020). Nevertheless, non-ionic surfactants are the second most extensively used surfactants in industries such as textiles, food, paper, and plastics. Triton X-100 (Wei and Liu, 2000), Tween 80 (Amaral et al., 2008), and cocodiethanolamide are the three most regularly used non-ionic surfactants (Ranjani and Ramamurthy, 2010b).

Composite foaming agent

Composite foaming agents or mixed surfactants have lately arisen as a promising source of synergistic interactions in the ongoing search for better surfactant characteristics. Varied researchers used various mixes of cationic, anionic, and non-ionic surfactants in their investigations (Amaral et al., 2008; Lunkenheimer and Malysa, 2003; Wang et al., 2014). It is claimed that a composite foaming agent made up of multiple types of surfactants produces better foam than the individual components (Khimani and Vora, 2011). It can also be hypothesised that in solutions having a mixture of surfactants, the tendency to form aggregated structures (mixed micelle) differs significantly from that of solutions containing only the pure surfactant (Harwell and Scamehorn, 1993).

2.3 Review on foam

Foam is formed by the juxtaposition of bubbles, resulting from gas dispersed from a liquid and the action that causes the formation of foam is called foaming. In the following sections, how the surfactant properties and the method of making of foam influence foam properties is discussed in detail. The walls of entrapped air or gas cells must be sufficiently tough and stable in the initial stages to preserve homogeneity of the mass until concrete hardens.

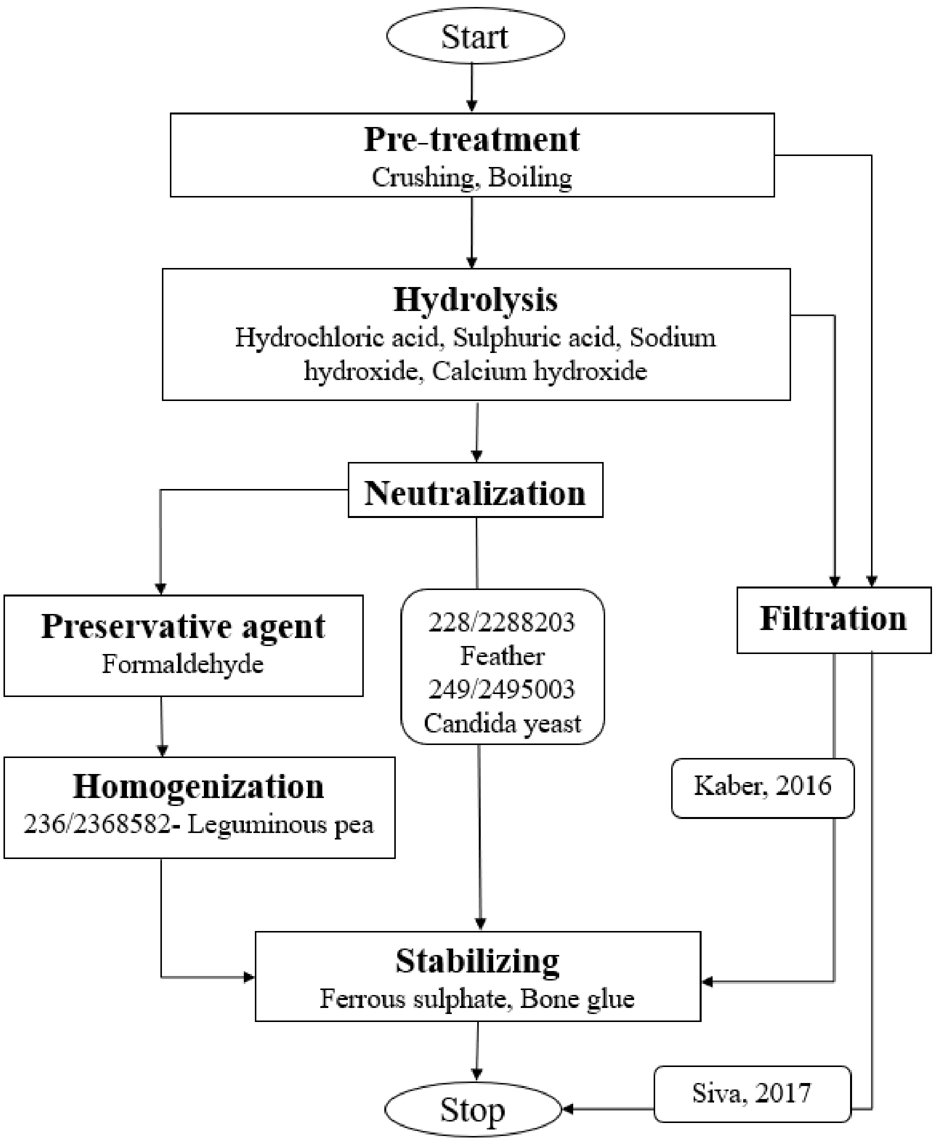


Figure 2-4 Manufacturing process of surfactant

2.3.1 Methods of Foam Production

Pure liquids cannot foam; therefore, foam generation and stabilisation require the presence of a surfactant (Birdi, 2009; Pugh, 1996). Most foam production systems comprise surfactant solutions, expansion gas (air), and equipment to combine, mix, and deliver the foam. One of the most conventional and well-known methods of creating foam is the dispersion technique, which involves either shaking or physically whipping a surfactant solution with a high-speed stirrer or fan (Krzan, 2013; Wei and Liu, 2000).

Researchers (Den Engelsen et al., 2002; Hirt et al., 1987) demonstrated that as the rotational speed of the stirrer is increased, the mean bubble size of the foam produced decreased. As a result, mixing speed is a crucial element for producing high-quality foam with this method. This method, however, is not mostly acceptable because precise control of the amount of air incorporated in the liquid is difficult to achieve. The aerosol method, which is used in pop drinks, involves supersaturating the liquid with gas by dissolving the gas under pressure into the surfactant solution and then releasing the pressure (Walstra, 1989). Due to its high cost, this method is not commonly used to produce foam. Foam is most commonly produced by mixing compressed air with a surfactant solution in high-density restrictions. This method is by far the most widely used method for creating foam for concrete. Research has demonstrated that foam produced using compressed air is relatively more stable due to its narrower bubble size distribution (Magrabi et al., 1999).

2.3.2 Foam Classification

Foam is usually classified based on its stability. They can also be classified according to their liquid fraction, as discussed below.

Wet foam and dry foam

Wet foam is typically defined as foam with a liquid fraction greater than 0.15. (Hutzler, Weaire, et al., 2005). A system with a value greater than 0.37 is referred to as extremely wet foam by Lykelama (Bergeron and Walstra, 2005). Generally, wet foam with a higher liquid fraction can be produced using dispersion and aerosol methods. As Aldridge (2005) suggested, wet foam can also be produced by spraying a foaming agent solution over a fine mesh. The foam produced by the aforementioned method has a large loose bubble structure with bubble sizes ranging from 2 to 5 mm and is relatively less stable (Barnes, 2008). As a result, such foam is not suitable for foam concrete production. On the other hand, as the gas fraction in foam increases, bubbles are forced to collide and form polyhedral structures, resulting in dry foam

(Bergeron and Walstra, 2005). The foam produced by the compressed air method is known as dry foam. In the compressed air method, dry foam is created by forcing a diluted solution through a series of high-density restrictions in a chamber with compressed air. Injecting pressurized air into the solution causes it to expand into a tight foam. In general, dry foam is relatively more stable and is recommended for foam concrete production. Extremely dry foam has a density of less than 0.05 (Hutzler, Weaire, et al., 2005).

Unstable, metastable and solid foam

As the name indicates, unstable foam lasts only for a short time before collapsing due to the overwhelming effects of surface tension and gravitational forces. These foams have a lifespan ranging from a few seconds to about 20 seconds (Lunkenheimer and Malysa, 2003). On the other hand, the metastable foam has a persistence ranging from a few seconds to months (Myers, 2020). They can withstand normal disturbances (Brownian fluctuations) but collapse when subjected to abnormal disturbances such as evaporation or temperature gradients (Kontogeorgis and Kiil, 2016). The most stable foam is the solid foam because of the presence of a mechanically rigid structure formed by an irreversible chemical process during or after foam formation. Most solid foams are created through processes of increasing temperature, releasing pressure, or chemical decomposition using blowing agents (which supply the gas) such as Fluorocarbons (currently prohibited in many applications), CO_2, and N_2.

2.3.3 Foam Properties and Review on Methods of Measurement

This section examines the important properties of foam, such as foam texture, foam expansion ratio, foam density, and foam stability along with their measurement techniques, to provide readers with information on their impact on the properties of foam concrete.

Foam texture

The shape and size of bubbles in the foam are referred to as foam texture (Osei-Bonsu et al., 2016). It has a big influence on the foam stability and foamability. The shape of foam bubbles can be characterised as spherical or polyhedral (Vijayaraghavan et al., 2009). The conductivity probe, the optical fibre-based approach, light transmission processes, and photographic methods, to mention a few, were all utilised in previous studies to investigate the bubble size distribution. Photographic approaches are said to be non-intrusive because they do not alter or distort the distribution of bubble sizes (Magrabi et al., 1999). Preformed foam commonly used in concrete has bubble sizes ranging from 0.1 to 1 mm (Wei et al., 2013). Numerous studies in the literature demonstrated that bubble size distribution is correlated with drainage rate (Magrabi et al., 1999). Without creating a stable framework, huge bubbles collided and rapidly merged into larger bubbles. Large bubbles exhibited greater deformability with no fixed shape during the flow. A stable foam cannot be created at a very low concentration of foaming agents; nevertheless, as the concentration of foaming agents increases, the foam's stability improves. In a porous medium, as the average diameter of bubbles lowers, the bubble becomes more homogeneous, resulting in tight grouping of foam (Li et al., 2019). Because of the reduced drainage, foam with smaller bubbles is said to be more stable (Miles et al., 1945). Furthermore, a more uniform bubble size distribution improves foam stability (Sheng, 2013; Sarma and Khilar, 1988). From the preceding observation, it is evident that fine-textured foam (small spheres of air) is necessary to produce foam concrete with a uniform and closed cellular microstructure (Brady et al., 2001). Hence studies on bubble size distribution in foam are needed to improve the properties of the foam and the microstructure of foam concrete.

Foam expansion ratio

The foam expansion ratio (FER) of a foaming solution measures its foam capacity or foamability (Vananuvat and Kinsella, 1975). Basically, FER is the ratio of the volume of foam formed to the volume of foaming solution used to generate the foam (Magrabi et al., 2002). The terms expansion ratio, foam multiplicity, or foam capacity are synonymous (Ospanova et al., 2014). Some researchers have employed foam scan equipment to assess foamability, based on the ratio of the volume of foam to the volume of gas injected for foam formation (Boos et al., 2013; Varade et al., 2011; Marinova et al., 2009). Surfactant characteristics and foam formation parameters, particularly surfactant concentration and FGP, have an impact on FER. As established by the foam density range prescribed by ASTM C 796, the foam utilised in the construction of preformed foam concrete should have FER in the range of 1:15.6 to 1:31.25. Foam with a very high foam expansion ratio larger than 0.05m^3 has a very thin lamellar layer, resulting in reduced stability. Over-expressed foams with low density are prone to collapsing, increasing concrete density (Ranjani, 2011). Foam with a lower expansion ratio, on the other hand, contains less air, resulting in inferior quality.

The foaming expansion ratio of the foam block produced based on the mixed foaming method increased as the w/c ratio increased from 50% to 70%, but it quickly decreased when the ratio exceeded 70%. Particularly, cracking occurred on the block when the w/c ratio was 80% because the viscous resistance of the slurry was inversely related to the w/c ratio. The viscous resistance of the slurry affected the gas expansion from the foaming agent (Gao et al., 2021).

Foam stability

Foam stability refers to the ability of foam to withstand spontaneous collapse or breakdown due to external causes. It reflects the life of the generated foam's lamellae (Laukaitis et al., 2005). Factors such as lamellae thickness, liquid viscosity, film elasticity, and bubble size (Wilde, 1996; Brown et al., 1953) have a substantial influence on foam stability. Further, foam stability is a critical requirement to produce foam concrete with a uniform cellular structure. Also, it is essential that the bubble must remain stable until the concrete sets. Various mechanisms are reported to influence the stability of foam such as gravitational drainage from films, coarsening caused by inter-bubble gas transport and coalescence of adjacent bubbles due to lamellar rupture (Tan et al., 2005; Murray and Ettelaie, 2004). Coarsening, or inter-bubble diffusion that causes larger bubbles to expand at the expense of smaller bubbles, is difficult to quantify (Magrabi et al., 2001). As a result, the drainage rate, which can be measured as free drainage or forced drainage, is frequently used to characterize the stability of foam (Hutzler, Cox, et al., 2005). Def-standard 42-40 (2002) has prescribed a methodology to assess the foam stability based on the free drainage technique for firefighting application Def- Standard 42-40. The guideline for firefighting foam states that the time for 25% drainage volume should not be less than 2.5 minutes (MIL-F-24385F, 1992). On the other hand, forced drainage is difficult to manage because the high pressure can cause bubbles to rupture. Foam stability has also been evaluated through studies on the variation of foam height, volume, or density over time (Lee et al., 2014; Sanova and Lisitsyn, 2012; Shrestha et al., 2007; Weschayanwiwat et al., 2005; Lunkenheimer and Malysa, 2003). Several studies have concluded that adding additives such as bone glue (Laukaitis et al., 2005) and the viscosity-enhancing agent could improve foam stability (Siva et al., 2015). Fine fillers, such as fly ash and powdered sand, have also been shown to increase foam stability by forming a homogeneous covering surrounding the lamellae and hence preventing coarsening (Awang et al., 2012; Nambiar and Ramamurthy, 2008b, 2006b).

Foam density

The unit weight of the foam is referred to as its density. Foam volume can be calculated using this parameter to achieve the desired density of foam concrete (Ranjani and Ramamurthy, 2010a). ASTM C796 recommends a foam unit weight range of 30-65 kg/m^3 for foam concrete, with the caveat that this range can be adjusted based on the foam chemical and generator used. It is also important to note that foam density is affected by a variety of factors such as surfactant type (Hamad, 2014), surfactant concentration (Ranjani and Ramamurthy, 2010a), additives (Çolak, 2000), foaming solution viscosity (Kroezen et al., 1988), lamellae thickness (Krzan, 2013; Gido et al., 1989), and FGP. The effect of foam production parameters on foam density, specifically surfactant concentration and FGP, will be discussed later.

A layer of liquid termed lamellae frequently surrounds the bubbles in the foam. Foam qualities are influenced more by the thickness of the lamellae and the liquid properties within the lamellae. The pressure in the bubbles requires a critical thickness of 5-15 nm, according to the researchers (Myers, 2020). High-density foam has a thicker lamellar layer than low-density foam. As a result, the foam stability is good when the thickness of the lamellar film around the bubble reaches the critical thickness. Few studies have shown that synthetic foam expands more and hence has a lower density than protein-based foaming agents (Amran et al., 2015). To enhance the density of low-density foams used in foam concrete, some foam thickening agents should be added as additives. Several studies have employed additions such as methylcellulose, sodium hydroxide, sodium carbonate, and sodium chloride to improve the viscosity of foaming solutions and hence the density of the foam (Siva et al., 2015; Ospanova et al., 2014; Çolak, 2000; Usman et al., 2012). As previously stated, improving foam concrete characteristics could be accomplished by adding additives which results in viscosity of the foaming solution and foam density (Siva et al., 2015).

2.4 Review on Foam Concrete Production

As discussed in section 1.1, depending on the foam production method, foam concrete can be classified as a pre-form or mixed foam concrete (Richard and Ramli, 2011). The impact of various foam and foam concrete production parameters on foam concrete quality will be discussed below.

FGP is one of the production parameters that controls the mixing of air and foaming liquid to manage foam quality. It's the pressure at which a foam generator produces foam. For a given surfactant concentration, foam density is said to be proportional to FGP. The size of the bubble in foam is also determined by the foam generating pressure. The microstructure of concrete is more consistently distributed when the foam is more stable and bubbles are smaller. Due to compressed air's inability to enter the foam solution, using an extremely low FGP resulted in poor foam quality. As a result, the cellular structure of foam concrete was compromised. In applications with very high FGP, the foaming solution also comes out with the foam as a result of turbulence, resulting in foam with high density and poor stability, as confirmed by a greater drop in density over time (Gowri and Anand, 2016; Siva et al., 2015). As a result, choosing the best FGP is critical because it affects foam density, bubble size, and, ultimately, the stability of foam and foam concrete mixes.

Surfactant concentration, which indicates the concentration of surfactant in the foaming premix solution, is another key foam formation parameter. It can also be stated as a dilution ratio, such as 1:x, in which one component foaming agent is diluted with x parts water. It was established in various studies that very low surfactant concentration created foam with extremely high density, but the foam produced was extremely unstable, as evidenced by its high drainage (Ranjani and Ramamurthy, 2010b). On the other hand, higher surfactant concentration resulted in a more viscous and stable foam, though, using higher surfactant concentration in foam concrete production is not cost-effective. As a result, selecting the best surfactant concentration to produce stable foam is necessary for cost-effective foam concrete production.

The type of mixer, batching and mixing sequences of foam concrete also significantly affect the quality of concrete. Several studies on the selection of mixer machines for use in preform foam concrete have been conducted by researchers. When a conventional mortar mixer with a blade rotating along the central axis is used, foam is found to float above the mix because of improper mixing. According to Karl and Worner (1994), the mixer should generate sufficient vertical motion of constituents to prevent the separation of light foam at the top and heavier ingredients at the bottom. Similar functional ability can be obtained with a food type mixer, commonly termed Hobart mixer with a helical or screw-type blade, as highlighted by Jones (2001). A pan mixer with rotating arms can also be one of the best mixing options. Its arm arrangement is adjustable and allows for clockwise and anti-clockwise rotation. Commonly, paddle-type mixer and the pan mixer are strongly recommended for preform production because the folding action protects the foam from the base mix. According to the limited literature on the mix foaming production method, a tilt drum mixer, a standard high-speed paddle type mixer, and a pan mixer may be suitable options for mix foaming concrete production (Akroyd, 2016). Further as per available literature, a horizontal propeller-type mixer with a 0.75-inch blade thickness and a rotating speed of 55 to 60 rpm is generally preferred for cellular concrete preparation. It should be noted that mixing speed and duration are important parameters because they affect foam stability (Brady et al., 2001).

2.5 Foam Concrete Properties

The properties of foam concrete are classified into fresh, mechanical, physical, durability, and functional properties. The fresh state of the foam concrete includes the mixture consistency, rheology and stability. In the hardened state, the main focus is on physical, functional and mechanical properties as described in the following sub-sections.

2.5.1 Fresh State Properties (Consistency and Stability)

Since foam concrete cannot be compacted or vibrated, it must have desired flowability and self-compatibility. These two properties are evaluated in terms of the consistency and stability of foam concrete, which is determined by the amount of water in the base mix, the amount of foam added, and other solid ingredients in the mix (Nambiar and Ramamurthy, 2008a). The marsh cone flow time and flow cone spread test are commonly used to determine the consistency of foam concrete (Jones and McCarthy, 2005c). These measurements were also linked to rheology, and it was discovered that coarse fly ash as a filler had a 2.5 times greater spread than cement sand mix when sand is replaced by fly ash on volume basis. The difference in aggregate particle shape and size is attributed to the improved consistency and rheology in the above study. Contrary to above in the same study, the consistency of the mix was reported to be reduced when sand is replaced by fly ash on mass basis. Adding to above high fluid consistency in the base mix and the high residual carbon in the ash, is reported to affect foam stability and necessitated larger foam volume to achieve the design plastic density (Jones and McCarthy, 2005c). Researchers discovered a decrease in mix consistency as the amount of foam in the mix increased. Reduced self-weight and greater cohesiveness resulting from higher air content and adhesion between the bubbles and solid particles in the mix increase the stiffness of the mix.

Generally, the state of stability is an adhesive behaviour of foam concrete mix design constituents and their consistency and cohesiveness as a system (Amran et al., 2015) which can result in the actual fresh density to be within ±50 kg/m^3 of the design density. Also, few researchers have tried other methods by checking the differences in actual and calculated w/c ratio (Jones and McCarthy, 2006), the measured and actual amount of foam (Awang and Aljoumaily, 2017; Jones and McCarthy, 2006) needed to achieve plastic density within ±50 kg/m^3 of design density. The water content required to generate a stable and practical foam concrete mix can be determined using regression equations developed for forecasting the spread flow value of foam concrete. A workability value of 45 percent is recommended to achieve foam concrete mix with desired stability and consistency as reported in various studies (Nambiar and Ramamurthy, 2008a).

From the perspective of fresh state concrete, the water-solids ratio is a very critical parameter which affects the consistency of concrete subsequently affecting the stability of concrete. So far, few researchers have studied the range of optimum water-solids ratio needed to achieve stable foam concrete mixes (Sahu and Gandhi, 2021; Jiang et al., 2016). Nambiar and Ramamurthy (2008a) studied the effect of the water-solids ratio on consistency and stability for mixes with different filler-cement ratios, fly ash replacement levels and foam volume. In this line, there are also earlier works reported on the effect of surfactant concentration (Narayanan and Ramamurthy, 2012) and the effect of the addition of viscosity-enhancing agent on the water-solids ratio (Sahu and Gandhi, 2021) requirement to achieve stable mixes.

2.5.2 Physical Properties

Density, drying shrinkage and air-void system are the major physical properties of foam concrete which contribute to its overall performance.

Density

The importance of foam concrete density is well documented (Jones and McCarthy, 2006; Nambiar and Ramamurthy, 2006a; Jones et al., 2003). One of the primary benefits of foam concrete is the ability to have wide range of densities (300 to 1850 kg/m^3). The properties of foam concrete are strongly influenced by designed densities. Despite the fact that there is no standard method for proportioning of foam concrete, the general criteria for w/c ratio, free water content, and maintaining a unit volume based on a defined goal of plastic density is the fundamental design criterion (Jones and McCarthy, 2006). It's difficult to design for a certain dry density because foam concrete desorbs between 50 and 200 kg/m^3 of total mix water, depending on the concrete plastic density. Plastic density tolerance is often acceptable up to ±50 kg/m^3 of the desired value, which is typical of foam concrete manufacturing practice (Jones and McCarthy, 2006). Densities in foam concrete mostly vary due to instabilities, segregation and collapse of foam.

Shrinkage

Although foam concrete was initially envisaged as an insulation material, there has been increasing widespread use in semi-structural and structural applications in the last few years. The substantial drying shrinkage, which has been reported to be up to 10 times larger than that of regular weight concrete, is one of the biggest deterrents to its structural application. Shrinkage is basically a cement paste-related phenomenon; for example, the shrinkage strain of cellular cement paste is as high as 0.5 percent, whereas the shrinkage strain for cellular cement-sand mixes generally varies from 0.1 to 0.35 percent. According to studies, the use of increased cement and water content, the lack of coarse particles in the mix composition, and the use of finer sand could all contribute to the higher volume of shrinkage in cellular concrete such as aerated concrete and foam concrete. Moreover, it is commonly accepted that shrinkage is a critical characteristic of concrete as it can lead to undesired cracking which is a common source of distress in concrete structures (Neville, 1995). Common classification of several forms of shrinkage as reported in literature according to their nature and time of occurrence are plastic shrinkage, drying shrinkage, autogenous shrinkage and carbonation shrinkage.

A review of literature in this context indicates that there are few studies conducted on the influence of parameters such as the effect of dilution ratio of foaming agent (Yu et al., 2011) volume and specific surface of micropores (Georgiades et al., 1991; Ziembicka, 1977), pore distribution and moisture content (Schubert, 1983), the crystallinity of hydration products (Alexanderson, 1979) on drying shrinkage of cellular concrete. However, the information available on the plastic, autogenous and chemical shrinkage of this lightweight porous material is very limited.

Air-void systems

It is well established fact in the literature that the pore structure of foam concrete is an essential feature because it impacts the other considerable properties like strength, permeation and durability. The pore system of foam concrete is classified in terms of gel pores, capillary pores, and entrained and entrapped air voids. Several researchers have shown that big capillary pores (50 to 10000 nm) and extra-large holes (>10000 nm) affect the strength and permeation characteristics of foam concrete, whereas small pores (less than 10 nm) and medium capillary pores (10 to 50 nm) affect shrinkage and creep (Khwairakpam et al., 2020; Jitchaiyaphum et al., 2013). Further few researchers have proved that porosity and pore size distributions vary considerably with the mix composition and curing method of foam concrete. So far, air void characterization studies on foam concrete have been conducted to determine the influence of foam volume (Nambiar and Ramamurthy, 2007a), filler type and fly ash replacement for fine aggregate (Hilal et al., 2015b; Nambiar and Ramamurthy, 2007a), mineral admixtures as cement replacement (Batool et al., 2019; Batool and Bindiganavile, 2017), type of surfactant (Panesar, 2013; Sahu et al., 2021), foam production parameters (Sahu and Gandhi, 2021), water to cement ratio (Jiang et al., 2016; Wee et al., 2011) and curing method (Dhasindrakrishna et al., 2021; Jiang et al., 2016). For instance, there is much evidence in the literature that foam concrete with larger foam volume shows wider air void distribution with larger pores than foam concrete with smaller foam volume (Liu et al., 2021; Sahu and Gandhi, 2021; Hilal et al., 2014b; Nambiar and Ramamurthy, 2007a). Few studies have proved that the inclusion of fly ash as filler helps in achieving a more uniform pore structure than fine sand (Nambiar and Ramamurthy, 2006b). In this line, mineral admixtures when used as cement replacement is also found to reduce pore size distribution in foam concrete (Batool and Bindiganavile, 2017; Hilal et al., 2014b; Nambiar and Ramamurthy, 2007b; Balendran et al., 2002; Gowripalan, 1990). Further few recent studies have proved that the different nature of foaming agents results in a different type of void structure in concrete (Sun et al., 2018; Falliano et al., 2018a; Tarameshloo et al., 2017; Panesar, 2013).

As the water-solids ratio exerts significant influence on the rheological properties of concrete, it will also affect the related properties such as pore structure, and the bubble maintaining the capacity of mortar. In this line, few studies have proved that the w/c ratio significantly affects the porosity and permeability of foam concrete (Hughes, 1985; Mehta, 1980). Limited studies on the influence of w/c ratio on pore structure have established that the adoption of smaller w/c ratio results in a higher volume of smaller dimension pores with thinner pore walls. On the other hand, the use of a higher w/c ratio resulted in pores with larger pore diameter and uneven pore size distribution (Gong et al., 2021; Liu et al., 2016). On a similar note (Jiang et al., 2016) reported that at a low water-solid ratio, the slurry has a strong ability to restrict the growth of air bubbles than at a high water-solid ratio. Thus, increase in the water-solid ratio improves concrete mobility and reduces yield stress and plastic viscosity which in turn contributes to an increase in air void size (Wee et al., 2011). Hence from the above discussion, it is evident that, as the w/c ratio affects the pore structure, it is expected to be an important controlling factor which governs the compressive strength of foam concrete. Further from the review of literature, it is observed that there are only limited studies reported on the relationship between permeability and pore characteristics of foam concrete (Hilal et al., 2014a; Kearsley and Wainwright, 2001b).

Characterization of air voids is most commonly accomplished by identifying air-void parameters such as void size distribution, shape factor, and spacing factor (Nambiar and Ramamurthy, 2007a). Foam texture represents the shape of the air voids. The shape factor measures how close the shape of the void is to a circle with an upper limit of 1 and a lower limit of 0; a value of 1 indicates a perfect circle, whereas collapsed or merged bubbles has reduced circularity value (Batool, 2015). It can be noticed that as the density decreases, the frequency of occurrence of such voids that are circular also decreases. It is observed that mixes with a narrower range of air-void size distribution showed higher strength. Whereas at lower density, which means higher foam content, air void structure has larger voids and wider distribution of voids, resulting in reduced strength (Nambiar and Ramamurthy, 2007a). The spacing factor influences the compressive strength of the cement-based foam and has an inverse relationship with the density (Wee et al., 2006). As the density increases, the distances between the voids get larger (Nambiar and Ramamurthy, 2007a).

Optical microscopy, scanning electron microscopy (SEM) and X-ray tomography (XRT) are the various image analysis techniques that are used to characterize air voids (Yu et al., 2010; Ungkoon et al., 2007). For SEM testing, imaging should be performed in a high vacuum of a conductive coating (gold). The accelerating voltage required for the scanning electron microscopy was 20kV (Yu et al., 2010). According to Batool (2015), SEM testing images should be magnified by150x to bring out a clear picture of the microstructure of voids and solid phases in cement-based foam. Later, image analysis software should be used to identify and quantify the air-void parameters, i.e., air-void size distribution, shape factor, and spacing factor for each mix.

For optical microscopy technique, an image analysis system should consist of an optical microscope and a computer with image analysis software. Nambiar and Ramamurthy, (2007a) investigated the structure of the macropores of foam concrete which are considered as pores having a diameter larger than 50 µm, a magnification of 20× was selected with a pixel representing 16 microns, and each image covering 44.23 mm^2 (7.68 mm × 4.23 mm). A total of 36 images should be captured for each mix from the six-cut surfaces (Sahu and Gandhi, 2021). XRT is a completely non-destructive method. In XRT, one can capture 3D images, but it has some specimen size limits due to the need for high-flux X-ray sources of narrow energy distribution, which increases with the size of specimen (Bentz et al., 2000).

2.5.3 Mechanical Properties

In general, the compressive strength of foam concrete is dependent on various parameters such as w/c ratio, type of sand, cement-sand ratio, type of foaming agent and foam production parameters, the volume of foam, curing method and characteristics of other additional ingredients added to the mix. Among the various parameters, the major controlling factor of compressive strength is the amount of air voids in the concrete which governs the density of concrete (Falliano et al., 2018b). Particularly for concrete mixes with lower density, the amount of entrained air voids controls the strength of concrete to the greatest extent (Falliano et al., 2018b; Tam et al., 1987). The w/c ratio is another controlling factor which governs the compressive strength of foam concrete. Few studies have reported that, unlike normal weight concrete where the w/c ratio is an important factor influencing strength, however in foam concrete, density controls strength more than that of w/c ratio (McCormick, 1967). Contrary to the above observations, Falliano et al. (2018a) observed that increasing the w/c ratio from 0.3 to 0.5 resulted in remarkable increase in strength from 0.3 MPa to 4 MPa which can be ascribed to the greater amount of hydration of cement. On a similar note, Piotrowski and Prochoń, 2018 studied the effect of variation of compressive strength of GBFS (granulated blast-furnace slag) based Geopolymer foam concrete with w/c ratio and established that there exists an optimum w/c ratio at which the strength is maximum. Further studies have reported that the compressive strength of foam concrete decreases with an increase in void diameter for dry densities between 500 and 1000 kg/m^3 (Kearsley and Wainwright, 2001c). In this line, Falliano et al. (2020), established that increase in w/c ratio can result in reduction of foam content requirement for a given target density. Not only did w/c ratio affects the compressive strength, but the amount of sand and fineness of sand also affects the pore structure and subsequently affects the compressive strength (Nambiar and Ramamurthy, 2008b; Nambiar and Ramamurthy, 2006b; McCormick, 1967).

Furthermore, the surfactants and the properties of the foam are also reported to have a manifest influence on the microstructure of concrete which in turn affects the strength. For instance, studies by researchers (Falliano et al., 2018a; Liu et al., 2019; Sun et al., 2018; Tarameshloo et al., 2017; Kuzielová et al., 2016; Panesar, 2013) proved that the stability of foam governs the pore size which consequently affects the strength of foam concrete. In this line, Falliano et al. (2018a), established that some protein-based surfactants which can result in foam with a higher fluid phase can facilitate more hydration of cement and thus enhance strength development than that synthetic surfactant. Furthermore, Falliano et al. (2018a) added that the relationship between the w/c ratio and compressive strength of foam concrete depends on the foaming agent type. Also, the addition of additives like CMC, xanthan gum and $Ca(OH)_2$ can enhance the stability of foam by aggregating the plateau border, enhancing the viscosity of surfactant solution which further results in the enhancement of the strength of concrete (Xiong et al., 2021; Hajimohammadi et al., 2018).

Another key factor influencing foam concrete's compressive strength is its curing method. In accordance with ASTM C 796, lightweight concrete samples and cellular concrete samples should be cured in a moist room with 100% relative humidity at least three days before compression testing is conducted. After the samples have been cured, they should be oven dried for 72 hours at 60°C. To achieve the desired compressive strength, Fujiwara et al. (1995) suggested that the samples to be cured in normal moist air for one day, and then in steam where the temperature should increase to 20°C/ h to preserve at 65°C for 4 hours and then cooled in air. When the cement was replaced with 50% fly ash, Kearsley and Visagie (1999) found a suitable curing temperature of 40°C to be likely to provide optimal performance. Falliano et al. (2018 a) compared the effect of 3 different curing conditions viz., water, air and cellophane sheets on the compressive strength of foam concrete. Based on their experimental outcomes, it was concluded that higher compressive strength was obtained in water curing and specimens sealed with cellophane sheets than that of air-cured specimens. A review of mixes used by different researchers and the obtained compressive strengths of foam concrete mixes is shown in Table 2-3.

Considering tensile behaviour, based on the literature review, it is evident that there has been very limited research reported on the effect of w/c ratio on foam concrete performance. Foam concrete is reported to display lower splitting tensile strength than comparable normal weight and lightweight aggregate concrete mixes, with higher values observed for mixes containing sand than those containing fly ash. A contributing factor to this increase is the improved shear strength between the sand particles and the paste (Jones and McCarthy, 2005a). The use of polypropylene fibres in concrete is reported to increase its tensile and flexural strength, provided the fibres do not affect the behaviour of freshly poured concrete or its self-compaction (Kearsley and Mostert, 1997). Researchers have reported flexural strength to compressive strength ratio of 0.25 to 0.35 for cellular concrete (Valore, 1954).

Modulus of elasticity

The static modulus of elasticity of foam concrete is said to be much lower than that of normal weight and lightweight concrete, with values ranging from 1.0 to 8.0 kN/mm^2, for dry densities between 500 and 1500 kg/m^3 respectively (Jones and McCarthy, 2005b). Normal weight concrete exhibited E-values that were up to four times greater than foam concrete of equivalent strength. According to reports, foam concrete that contains fly ash as fine aggregate has a lower E-value than foam concrete that contains sand. The difference between sand and fly ash mixes is due to the proportion of fine aggregate in sand mix versus paste in fly ash mix (Jones, 2001). The E-value has been found to increase by two to four times when polypropylene fibre is used (Jones and McCarthy, 2005b).

Table 2-3 Summary of compressive strength reported

Author(s), year	**Mix details**	**Density range kg/m³**	**Comp. strength MPa (28 days)**
McCormick (1967)	S-C: 0.79-2.80; w/c:0.35-0.57; Cement: 335-446 kg/m³	800-1800	1.8 – 17.6
Tam et.al (1987)	S-C: 1.58-1.73; w/c: 0.6-0.8; Cement: 390 kg/m³	1300-1900	1.81 – 16.72
Regan & Arasteh (1990)	LAC-C: 0.6, w/c: 0.45-0.6	800-1200	4 – 16
VanDeijk (1991)	Cement –sand/flyash	280-1200	0.6 – 10 (91-d)
ACI523.1R-1992	Neat cement: paste	240 – 640 (DD)	0.48 – 3.1
	Cement-sand mix	400-560 (DD)	0.9 – 1.72
Hunaiti (1997)	S-C: 3	1667	12.11
Kearsley & Booyens (1998)	Cement-flyash replacement	1000-1500	2.8 – 19.9
Durack & Weiquing (1998)	S-C:1.23-2.5; w/c: 0.61-0.82; Cement: 270-398 kg/m³	982-1185 (DD)	1 – 6
	F-C: 1.48-2.5; w/c: 0.48-0.7; Cement: 137-380 kg/m³	541-1003 (DD)	3 – 15 (77-d)
Kearsley & Wainwright (2002)	Cement-fly ash replacement Cement: 193-577 kg/m³, w/c: 0.6-1.17	1000-1500	2 – 18
Tikalsky et. al. (2004)	C; Cement: 149-420 kg/m³; w/c: 0.4-0.45	490-660	0.71 – 2.07

Author(s), year	**Mix details**	**Density range kg/m^3**	**Comp. strength MPa (28 days)**
	Cement-sand/fly ash; Cement: 57-149; w/c: 0.5-0.57	1320-1500	0.23 – 1.10
Aldridge (2005)	Cement-sand mix	400-1600	0.5 – 10
Jones & McCarthy (2005a)	S-C: 1.83-3.17, w/c: 0.5; Cement: 300 kg/m^3	1000-1400	1 – 2
	F-C: 1.22-2.11; w/c: 1.11-1.56	1000-1400	3.9 – 7.3
Jones & McCarthy (2005b)	S-C:1.5-2.3, w/c:0.3; Cement: 500 kg/m^3	1400-1800	10 – 26
	F-C:1.15-1.77, Cement: 500 kg/m^3	1400-1800	20 – 43
Nambiar & Ramamurthy (2006)	Cement-sand mix (coarse sand)	800-1350(DD)	01 – 07
	Cement-sand mix (fine sand)	800-1350(DD)	02 – 11
	Cement-sand /fly ash mix	650-1200(DD)	4 – 19
Kim et al. (2012)	Cement – quartz	400-1500 (DD)	1.5 – 29.2
Bing et al. (2012)	Cement – flyash- SP-PP/ SF	800-1500	10 – 50
Chen et al. (2013)	Cement – flyash- SP-PP/ SF	400-800	1.58 – 11
Roslan et al. (2013)	Cement- flyash- lime-PP	1400	5.4 – 13.2
Hilal et al. (2015b)	Cement- sand/ flyash _SF-SP	1300-1900	19 – 48

Author(s), year	Mix details	Density range kg/m³	Comp. strength MPa (28 days)
Kramer et al. (2015)	Cement sand-SP, w/c: 0.5	1000-1100	7.9 – 10.6
Gowri & Anand (2018)	Cement-flyash, w/c:0.4	1600	16.24 – 23
Zhang et al. (2022)	Cement-flyash-slag-SF	1000-1300	9.6 – 15.6

S-C: sand-cement ratio; F/C: flyash-cement ratio; w/c: water-cement ratio; a/c: ash cement ratio; LAC: lightweight aggregate content, SF- silica fume, GGBS- Ground Granulated Blast-furnace Slag, SP: superplasticizers, PP: polypropylene fiber; DD: dry density: d- days

2.5.4 Durability Properties

Permeation Characteristics

Foam concrete more readily takes up moisture from the atmosphere than dense concrete. From the review of literature, it is observed that there are only limited studies reported on the relationship between permeability and pore characteristics of foam concrete (Hilal et. al., 2014, Kearsley and Wainwright, 2001). Permeation characteristics of concrete are commonly measured in terms of permeability, water absorption and sorption. Water absorption is the basic measurement test commonly adopted to assess the water permeation based on the total volume of pores in concrete. On the other hand, sorption which gives the index of moisture movement into concrete based on capillary action is considered as the realistic method as it reflects the way most concrete is penetrated by water (Dias, 2004). Due to greater difference in density, expressing water absorption as a percentage by mass can produce misleading results in foam concrete. In this line studies have shown that it is preferable to express the water absorption as % by volume. Studies conducted by Kearsley and Booysens (1998) have proved that increase in porosity and fly ash content resulted in increase of foam concrete's oxygen and water vapour permeability. The permeability coefficient of lightweight foam concrete is related to unit weight and inversely proportional to pore ratio (Byun et al., 1998). Earlier studies on permeation characteristics have proved that entrained air voids can be considered as aggregates in foam concrete and their inclusion reduces the permeability by increased tortuosity and reduced microcracks at the interface of air void and mortar (Hilal et al., 2014a). On a similar note, studies by Nambiar and Ramamurthy (2007a) have proved that water permeation characteristics reduced with density of foam concrete due to reduction in capillary pore volume in mixes with reduced paste content, confirming that all artificial air pores are not taking part in permeation. However, contrary to above observations, few studies have proved that oxygen and water vapour permeability of foam concrete is found to increase with increasing porosity (Kearsley and Booysens, 1998). Hence water movement into foam concrete is a complex function of various parameters such as pore diameter distribution, pore connectivity, tortuosity and porosity (Nambiar and Ramamurthy, 2007a). Limited studies have also been made on the influence of filler type and fly ash replacement for fine aggregate (Jones and McCarthy, 2005c), use of mineral admixtures (Nambiar and Ramamurthy, 2007a; Kearsley, 1999), influence of w/c ratio, type of surfactant (Sun et al., 2018), addition of additives to surfactants (Sahu and Gandhi, 2021) on the water absorption and sorption characteristics of foam concrete. Sorptivity of foam concrete is reported to be lower than that of the corresponding base mixes, and the values are trend to decrease as foam volume increases (Nambiar and Ramamurthy, 2007a; Madjoudj et al., 2002; Giannakou and Jones, 2002). When the sorptivity of foam concrete mixes using sand and fly ash as aggregate was compared, it was discovered that mixtures containing fly ash had a somewhat higher sorptivity than mixes containing sand. On a similar note, Nambiar and Ramamurthy (2007a), reported that use of fly ash as sand replacement resulted in increase in water absorption of foam concrete. On the other hand, use of mineral admixtures such as fly ash as replacement for cement resulted in lesser water absorption due to the modification of pore structure (Nambiar and Ramamurthy, 2007a; Kearsley, 1999). Furthermore, few recent studies have proved that addition of stabilizer to improve the foam quality have shown improvement in pore structure and consequently resulted in reduction in water permeation characteristics (Xiong et al., 2021). In this line, studies by Panesar, 2013, have proved that type of surfactant also has significant influence on water permeation characteristics of foam concrete.

Resistance to aggressive environment

The response of foam concrete to aggressive environment is dependent on the size and volume of pores, the distribution mechanism, and the composition of the mixture. It has been shown that the foam concrete's resistance to aggressive environment depends largely on its cell-like structure, which does not necessarily make it less penetration-resistant to moisture compared to normal concrete; the air-voids reveals to act as a buffer preventing rapid penetration (Nambiar and Ramamurthy, 2007b). In the course of its service life, foam concrete may be affected by substances like sulfate. Sulfate attack is complex and is influenced by several factors including cement type, mineral admixtures, cations associated with sulfate anion, permeability, sulfate concentration, time and duration of exposure (Brown et al., 2004; Cao et al., 1997; Prasad et al., 2006; Sahmaran et al., 2007). Few literature evidences indicate that, foam concrete has a great resistance against sulphate and carbonation attacks. Ranjani and Ramamurthy (2012) investigated the behaviour of foam concrete with densities ranging from 1000 to 1500 kg/m^3 in solutions of 0.5 percent and 5 percent sodium sulphate and 0.424 percent and 4.24 percent by mass magnesium sulphate for a year. The expansion of foam concrete in a sodium sulphate environment was 28 percent higher than in a magnesium sulphate environment, owing to a higher rate of ettringite formation in the sodium sulphate environment, but the major deterioration mechanism in the magnesium sulphate environment was due to disintegration of cementing material, which contributed to a 1 percent mass loss of samples (Ranjani and Ramamurthy, 2012). When compared to base mix samples (mortar without foam) the resistance of foam concrete to sulphate environment was found to be good. A few researchers looked at the carbonation resistance of foam concrete. Jones et al., (2003) discovered that replacing fly ash with cement in the mix resulted in stronger carbonation resistance than mixes with fine sand. Furthermore, a higher volume of foam content was discovered to diminish carbonation resistance as the density decreased (Jones and McCarthy, 2005b, c). Jones and McCarthy (2005a, b) also found that lower density concrete carbonates at a faster rate than greater density concrete. Hence in this line Jones and McCarthy (2005a) proved that while designing low-density concretes, several parameters impacting the foam concrete should be considered, including water adsorption rate, depth of primary penetration, and degree of sufficient resistance to freezing and thawing phenomena. On the contrary, few studies have proved that a low-density design protected the foam concrete from corrosion better than a higher-density design (Chindaprasirt et al., 2008; Newman and Choo, 2003; Kearsley and Booysens, 1998). In terms of chloride intrusion, it was observed that the resistance of foam concrete to chloride ingress was comparable to that of conventional concrete with a compressive strength of 25 MPa. On a similar note, limited studies on alkali-silica reaction damage of foam concrete have proved that damage of alkali silica reaction damage was not significant despite using recycled aggregate.

2.5.5 Functional Properties

Thermal Insulation

Due to its cellular microstructure, foam concrete has excellent thermal insulating properties. The thermal conductivity of foam concrete density of 1000 kg/m^3 is reported to be one-sixth of the value of a typical cement-sand screed (Aldridge and Ansell, 2001). Richard et al. (1975) found that it enables speedy and settlement-free construction with strong thermal insulation, excellent freeze/thawing qualities, and good fire resistance. Sound and thermal insulation of floors, thermal protection of flat, mono-pitched, and double-pitched roofs are some of the thermal related applications of foam concrete. According to Giannakou and Jones (2002), a foam concrete ground supported slab foundation is thermally efficient, has good permeability qualities, and provides enough strength. Thermal conductivity values range from 0.1 to 0.7 W/mK for dry densities ranging from 600 to 1600 kg/m^3, with values decreasing with decreasing densities (Jones and McCarthy, 2005a). When the inner leaf of a brick wall is replaced with foam concrete with a unit weight of 800 kg/m^3, the insulation can be increased by 23% (Taylor, 1965). Hence, insulation is roughly inversely proportional to concrete density (Shrivastava, 1977). A reduction in concrete dry density of 100 kg/m^3 results in a reduction in thermal conductivity of 0.04 W/mK of lightweight aggregate foam concrete (Weigler and Karl, 1980b). Changing the mortar/foam ratio affects density, which significantly impacts insulation capacity (VanDeijk, 1991). Giannakou and Jones (2002) demonstrated the effect of replacing cement with

Pulverized Fuel Ash (PFA) on concrete thermal conductivity. Thermal conductivity of foam concrete with 30% PFA was reduced by 12 to 38% compared to mixes with only Portland cement as a binder. This decrease in thermal conductivity is attributed to fly ash particles lower density and cenospheric particle morphology, which increases the heat flow path. Weigler and Karl (1980b) investigated the use of lightweight aggregates (LWA) with low particle density in mortar matrix with artificially introduced air voids and proved that LWA are beneficial in lowering thermal conductivity. Proshin et al. (2005) discovered that by moderately filling porous mortar with polystyrene granules, foam concrete with densities ranging from 200 to 650 kg/m^3 and thermal conductivities ranging from 0.06 to 0.16 W/mK can be produced. Further, thermal insulation is said to improve as the temperature drops (Richard, 1977). While researching the possibilities of cellular concrete for load-bearing insulations for cryogenic applications, Richard et al. (1975) reviewed the thermal and mechanical properties of foam concrete. Various densities ranging from 640 kg/m^3 to 1440 kg/m^3 were impacted by temperature fluctuations ranging from 22°C to -196°C. When the temperature was reduced from 22ºC to -196ºC, the thermal conductivity of foam concrete was reduced by 26%. Tada (1986) has proposed an optimal material design based on the thermal performance requirements for building.

Acoustical properties

Recent studies have shown that foam concrete is more acoustically efficient than regular concrete because of its cell-like microstructure (VanDeijk, 1991; Taylor, 1965; Narayanan and Ramamurthy, 2000). The sound insulation resistance or reflected frequency volume, on the other hand, is mostly governed by the stiffness of the concrete mass (surface density). The frequency of sound reflection is presumably dependent on the thickness of the wall and its bulk density, according to the theory of solid wall sound resistance (Tada, 1986). As a result, when a concrete wall is extremely stiff, it can reflect sound more effectively than a cellular wall that can absorb it (Laukaitis and Fiks, 2006). The frequency of sound transmitted by the foam concrete cellular wall is up to 3% greater than that of a typical concrete wall. When compared to dense concrete, foam concrete offers a 10 times higher rate of sound absorption (Valore, 1954; Jones et al., 2003). The amount, size, and distribution of pores, as well as the consistency of the foam concrete, can affect its sound absorption (Jones and McCarthy, 2005c; Narayanan and Ramamurthy, 2000; Jones and McCarthy, 2005b).

Fire Resistance

At high temperatures, radiation influences heat transfer through porous material, which is an inverse function of the number of solid air interfaces traversed. Foam concrete may have increased fire-resistant capabilities due to its reduced thermal conductivity and diffusivity (Valore, 1954). Different densities of foam concrete were tested for fire resistance, and it was shown that reducing density enhanced fire endurance. Jones and McCarthy (2005b) concluded that the proportionate strength loss on fire exposure for lower densities of foam concrete was reduced when compared to conventional concrete. Foam concrete with lower densities has proved to be more fire resistant than vermiculite concrete, however larger densities have the opposite effect (Aldridge, 2005). Kearsley and Mostert (2005) investigated the influence of cement composition on the fire resistance behaviour of foam concrete. They discovered that hydraulic cement with an Al_2O_3/CaO ratio greater than 2 may survive temperatures as high as 1450^oC without harm.

2.5.6 Chemical Characteristics

Literature review reveals that very limited studies are available on microstructure and chemical analysis of foaming agents and foam concrete. This section discusses about the chemical characteristics along with techniques adopted for determination of same. Chemical characterization of cement paste is generally analyzed with X-ray diffraction (XRD), Energy Dispersive X-ray spectroscopy (EDS), Fourier Transform Infrared (FTIR) spectroscopy. EDS helps to determine the individual atomic composition of elements in the mix (Ungkoon et al., 2007). XRD gives knowledge about the structure of the cement paste and crystalline matter on an atomic scale (Ungkoon et al., 2007). However, XRD cannot detect the presence of the amorphous phase. Still, it can be quantified indirectly by the Quantitative XRD method (De La Torre et al., 2001). According to Batool (2015), specimen preparation was done by finely grinding the specimen to attain size of 25- 35µm. Ranjani and Ramamurthy

(2012) prepared the specimen by grinding it by hand and passing it through a 75 μm sieve. Other experimental specifications to be adopted at the time of study as reported by various researchers were as follows. Gualtieri et al. (2006) has reported that during the experiment Cu K_α Radiation was used in the range 5–80° 2θ using a laboratory Philips PW1729 Bragg–Brentano diffractometer equipped with a gas proportional detector. The counting time was 5 s for each 0.02° 2θ step. According to Ranjani and Ramamurthy (2012), for XRD analysis, Cu Kα radiation with a wavelength of 1.54 Å at a voltage of 30 kV, scanning speed of 1/min, and current of 30 mA was adopted. The obtained results in the diffraction patterns will explain the chemical composition (Yu et al., 2011).

The Rietveld Method, the most extensively used approach, was utilised to quantify the detected phases using X-ray diffraction (QXRD). The Rietveld method yields the sum of the phases fraction normalised to 100 percent crystalline phases, with the amorphous material often left out. The Rietveld refinement utilising the pseudo-Voigt peak shape function was performed using a publicly available software package called Generalized Structure Analysis System (GSAS) (Gualtieri et al., 2006). GSAS can refine up to nine phases at once and requires structural models for all phases as input, which can be found in the American Mineralogist Crystal Structure Database (AMCSD). The "Chi^2" factor 90-which is the Rietveld Discrepancy Value is considered as important criteria for judging the goodness of fit (Toby, 2006).

For the investigation of hydration products in cement paste, several authors used FTIR, thermal analysis, thermogravimetric analysis (TGA), scanning electron microscopy (SEM), EDS, and XRD techniques. For instance, Horgnies et al. (2013) studied cementitious materials using FTIR spectroscopy. Various mineral phases in cementitious materials such as portlandite [Ca $(OH)_2$] and Calcium Silicate Hydrate (C-S-H) were identified through this study. Yuanliang et al. (2021) used SEMEDS and TG/DSC to investigate the changes in the composition of foaming agent due to addition of foam stabilizer. In this line Siva et al. (2017) studied FTIR spectra of foaming agents to evaluate the structural arrangement and to study the impact of surfactant extraction technique on surfactant microstructure. Sahu and Gandhi (2021) based on the EDS analysis results showed that base mixes (without foam) have higher

Ca/Si ratio when compared to foam concrete mixes (Ungkoon et al., 2007; Wei et al., 2011).

2.6 Major Gaps in Research Work

Review of literature reveals some important facts regarding foam concrete which are discussed below. Synthetic surfactants commonly used in foam concrete production is reported to exhibit serious environmental threat. Hence utilization of natural surfactants could present new sight in improving the environmental impact of foam concrete. However, studies on valorization and extraction of natural surfactants from different plant and animal sources for use in foam concrete is very scarce from Indian context. In spite of the fact that foam concrete offers several advantages over conventional concrete, its use in India is relatively low when compared to foreign countries. Also, from the knowledge search it is found that foam concrete presently under research are mainly intended for non-structural applications such as void filling, thermal insulation, use for partition wall etc. Poor microstructure, higher drying shrinkage, higher water demand requirement, incompatibility with various chemical admixtures has limited its use only for non and semi-structural applications. Now with increasing interest in extending its use for structural applications, more systematic research is demanded for structural use. Among the various factors influencing the mechanical behaviour, water-solids is recognised as an important factor. Nevertheless, according to the review of the literature, it is evident that although the number of publications in the research area of foam concrete is steadily increasing, to date only 5% of total number of papers are reported on the effect of the water-solids ratio on the fresh state behavior, microstructure and mechanical behavior of foam concrete. This highlights the major research gap and necessitates systematic studies in this context. Further, review reveals that very limited studies are available on microstructure and chemical analysis of foaming agents and foam concrete.

2.7 Motivation for the Research

Considering the rapid growth of environmental alarm about the usage of synthetic surfactant, the current study aims to use natural surfactants valorized and extracted from two different biological sources, namely sesame seed (rich in protein) and hingot fruit (rich in saponin) in foam concrete production.

Balanites aegyptiaca also known as hingot is found all over the world, from tropical Africa to Myanmar. Studies on phytochemistry of various parts of plant indicates that fruit, seeds and bark part of plant has 22-27% of saponin (Oleszek and Hamed, 2010). The combination of the nonpolar sapogenin and the water-soluble side chain causes a saponin to foam, which is comparable to the structure of most synthetic surfactants with lipophilic and hydrophilic molecular components. Adding to above, excellent plant's adaptability, high yield features and abundant availability are other added reasons for selection of this plant for this study as it can add economical as well as environmental benefits. There are also literature evidences which shows that since ancient time, hingot have been used as detergent (Hall and Walker, 1991). Recently, few studies have highlighted its potential in the production of surfactants and cleansers (Barminas et al., 2016; Gour et al., 2015; Manji et al., 2013) for various applications. Currently, though hingot has been of various traditional uses, but scientific research works to ascertain its potential in domestic and industrial applications is scarce.

Another plant source used for surfactant extraction in the present study is *Sesamum indicum* commonly known as sesame seed. Although it is commonly grown for seed and oil in many regions of the world, the International Plant Genetic Resources Institute classifies it as an underutilised or neglected crop (IPGRI). Studies have proved that sesame seed has rich protein content of 20 to 25% (Singharaj and Onsaard, 2015) and hence has excellent potential foamability. The basic foaming mechanism of protein-based surfactants is that it creates air bubble due to breakage of peptide linkage and large protein molecules into more hydrophobic small molecules eventually resulting in reduction of surface tension of solution (Panesar, 2013). Despite the huge potential foamability of above surfactants (sesame and hingot), it is surprising to find that its potential has not assessed for use in foam concrete production. Studies on relative behaviour of concrete produced using these surfactants will present a new sight in improving the environmental impact of foam concrete.

Another important aspect of this project is to conduct an extensive experimental examination of the effect of the water-solids ratio on the various features of foam concrete, which has received very little attention in the literature. Hence in the present study, for a variety of densities, the impact of the water-solids ratio on various physical, chemical, and mechanical aspects of foam concrete has been examined. Further this is probably the first in-depth study on relative behaviour of foam concrete produced using aforementioned surfactants. The optimal water-solids ratios established for various mixtures can be used as a reference for future foam concrete performance research. Further, recommendations derived from this study could gradually boost the market's use of recommended natural surfactants, perhaps opening up new avenues for enhancing foam concrete's environmental impact.

2.8 Summary of Literature Review

This chapter highlighted the arduous requirement to identify locally available natural based surfactants as prospective supplementary alternative to conventional synthetic foaming agents for enhancing foam concrete's environmental impact. Natural surfactants are becoming more popular nowadays, because of its special attributes such as biodegradability, low toxicity, biocompatibility, low cost, specificity, high selectivity and activity even in severe environment with high pH, temperature, and salinity. Hence, identification of locally available cost-effective natural surfactants can facilitate extensive usage of foam concrete in India because of various economical as well as environmental benefits associated with it.

Surfactant properties significantly impact foam properties because they affect surface tension and gas-liquid interfacial properties. Further researchers have proved that foam produced with a more viscous surfactant solution has a high initial foam density and long-term stability. Hence, the selection of surfactant and the properties of foam are supposed to have manifest influence on the microstructure of concrete. In spite of their significant expected influence, the investigations on the influence of characteristics of surfactant and foam on the concrete properties are very limited.

Considering the fresh state behaviour requirements, as foam concrete cannot be compacted or vibrated, it must have desired flowability and self-compatibility which can result in stable mix. Researchers have derived the spread requirement of foam concrete mixes which can result in stable and consistent mixes. As the water-solids ratio exerts significant influence on rheological properties of concrete, it also affects the related properties such as pore structure and the bubble maintaining capacity of mortar. Limited studies on influence of w/c ratio on pore structure have established that adoption of smaller w/c ratio results in higher proportion of smaller pores with thinner pore walls and more connected pores. On the other hand, use of higher w/c ratio resulted in reduced pores with increased pore diameter and uneven pore distribution. Hence from the above discussion, it is evident that, as the w/c ratio affects the pore structure, it is expected to be an essential controlling factor that governs the compressive strength of foam concrete. In this line studies have proved that foam concrete with uniformly distributed spherical air voids showed higher compressive strength whereas irregular perimeter or combined form bubbles with large uneven opening exhibited lower compressive strength. Additionally, as shrinkage is a cement paste-related phenomenon, foam concrete, which is essentially a mortar, has a far higher shrinkage strain than regular weight concrete. But by using light-weight aggregates, high-range water- and shrinkage-reducing admixtures, fibres, and adopting the autoclave technique of curing, this might be diminished. In accordance with its microstructural features, foam concrete is said to have superior thermal and sound insulation properties. Additionally, research have shown that foam concrete with closed cells can have strong resistance to hostile environments because air spaces function as a buffer, delaying quick penetration.

Chapter 3

Objectives and Scope

3.1 General

From the review of earlier research and development in the field of foam concrete, the need for in-depth studies on the identification of locally available natural foaming agent, effect of water-solids ratio on foam concrete behaviour, chemical analysis of foaming agent and foam concrete have been highlighted. The objectives and scope for present study have been defined as outlined in the following sections.

3.2 Objectives

This study's overarching primary aim is to use the locally available natural-based surfactants in the foam concrete production. Accordingly, the following objectives were set out of course to this investigation:

i) To optimise the process of extraction of surfactant solution from sesame seed (*Sesame indicum*) and hingot fruit (*Balanites aegyptiaca)* and to evaluate the relative performance of foam produced with aforementioned natural surfactants through statistically designed experiments.

ii) To carry out extensive experimental investigations on the influence of composition of foam concrete (type of surfactant, foam volume, water-solid ratio) on foam concrete properties such as consistency, compressive strength, split tensile strength, water absorption and microstructure of foam concrete.

3.3 Scope

This work explores locally available natural-based raw materials in foam concrete production as an alternative to synthetic-based surfactants. Therefore, this work's scope is restricted to natural surfactant sources, viz sesame seed and hingot fruit. A constant cement-sand ratio of 1:2 and foam concrete design fresh density ranging from 640-1800 kg/m^3 have been adopted. Ordinary Portland cement 43 grade conforming to IS 269 and river sand finer than 300 μm is used for the entire research work.

3.4 Design of Research

The current study is conducted by adopting the following methodology as depicted in Figure 3-1.

- An in-depth review of various plant-based surfactants rich in protein and saponin is carried out. Further, a review on the methodology of extraction of surfactant from protein and saponin-based natural sources is also conducted.
- The optimization of surfactant extraction and foam production processes are carried out systematically based on RSM using Design Expert software. The parameters considered concerning the extraction process of sesame and hingot surfactants and foam production are surfactant concentration, temperature and duration of heating and FGP. However, for sesame surfactant extraction process, adding to above, sodium hydroxide concentration (alkali for hydrolysis) is also considered as additional parameter. The optimum levels of surfactant extraction parameters and foam production parameters will be derived by evaluating essential characteristics such as foam density, foam stability and viscosity of surfactant solution. Also, the correlation between various surfactant and foam properties such as viscosity of surfactant solution, foam density, foam drainage and foam bubble size are established Further ascertainment of the performance of these natural surfactants at the optimized parameters will be carried out based on the assessment of foam cement paste as per ASTM guidelines.
- Extensive experimental investigations on the influence of composition of foam concrete (type of surfactant, foam volume, water-solid ratio) on various foam concrete properties will be carried out. Firstly, the influence of composition on the fresh state properties (stability and consistency) of foam concrete for mixes with different foam volumes and for two different natural surfactants namely sesame and hingot is studied. This is followed by studies on the pore structure and the corresponding impact on the mechanical behaviour and water permeation characteristics of foam concrete. The optimum water-solids ratio

at which the fresh concrete characteristics, mechanical properties, and water permeation characteristics are satisfactory are derived for various densities of foam concrete. Further studies on microstructure are used to justify the experimental results

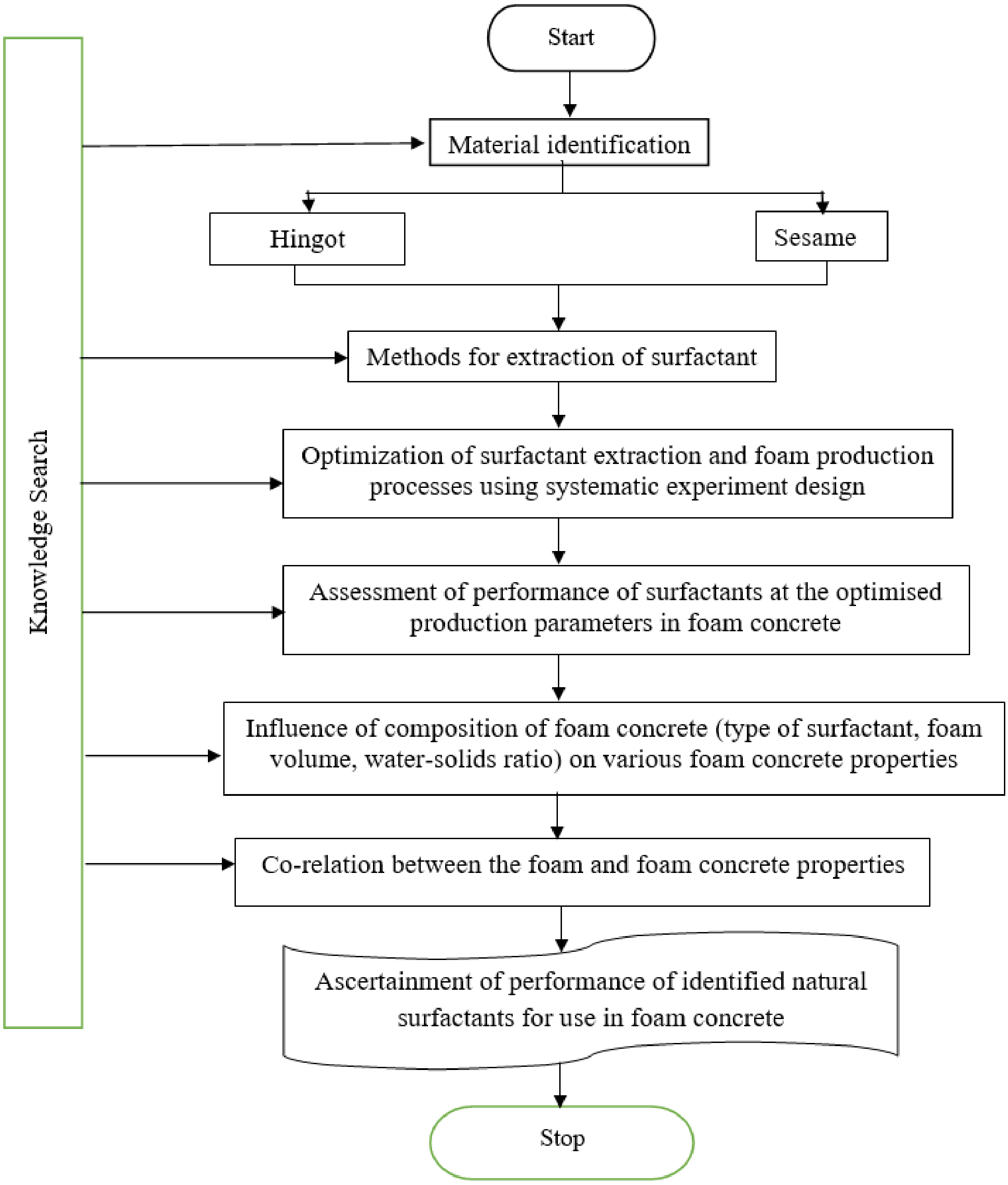


Figure 3-1 Proposed methodology

Chapter 4

Optimization of process of surfactant extraction from of *Balanites Aegyptiaca and Sesamum Indicum*

4.1 General

The ever-increasing environmental concern about synthetic surfactants triggers an interest in natural surfactant. It is to be noted that the synthetic surfactants commonly used in foam concrete production is reported to exhibit serious environmental threat in forms of chronic and sub-lethal toxicity in aquatic animals (Gour et al., 2015; Lewis, 1991), release of carcinogenic chemicals (Reddy et al., 2013), harmful impact on environment and human health due to surfactant residue in soil. To address the above environment concerns, natural surfactants are proposed as alternative to synthetic (Gour et al., 2015). In this context, many researchers are working on valorization and extraction of natural surfactants from different plant and animal sources. In India, there are many plant-based sources which are either rich in protein or saponin with potential foamability. Although many instances of natural surfactants have been documented in literature for different applications, however, most of the information on surfactants is available as patent documents, and only minimal information appears in published academic papers. Also, important thing to be noted is that, for a successful extraction process of surfactant, careful consideration of several variables such as the particle size of raw materials, the type and cost of extraction, the ratio of solid/liquid, extraction temperature and duration is needed (Jassal et al., 2016; Köse and Bayraktar, 2016; Yisong et al., 2022). In line with the above discussion, the present study attempts to assess the potential of surfactant extraction from hingot fruit (rich in saponin content) and sesame seed (rich in protein content) for use in foam concrete.

Also, when new foaming agents are developed, optimization of surfactant extraction process and foam production parameters is critical as the quality of foam and surfactant affects the concrete quality significantly. Hence the present study comprises of investigations conducted experimentally and statistically to optimize the surfactant extraction and foam production processes using Design Expert software. Further the suitability of foam in production of concrete will be assessed based on performance of foam cement paste in terms of strength and water absorption as per ASTM requirements.

4.2 Materials and Equipment used

For the present study, sesame seed are collected from the local market in Amingaon, Assam, India, while hingot fruit is collected from Madhya Pradesh, India. The details of extraction procedure of surfactant solution from sesame seed and hingot fruit is explained in detail in the author's previous publications (Khwairakpam and Gandhi, 2021, 2020). For both sesame and hingot surfactant extraction, pre-treatments steps including drying and particle size reduction are carried out to increase the mass transfer efficiency of the extraction. Further, to extract maximum foaming solution, hydrolysis is carried out wherein, powdered sesame seed diluted in water at a particular concentration is treated with NaOH solution and then subjected to heating in magnetic stirrer. Followed by hydrolysis, neutralization is carried out with ammonium chloride. Few studies had established that as the dissociation degree of ionized groups of protein is dependent on the pH of the solution and hence it is not recommendable to choose a highly acidic or basic solution. In this line, few researchers have recommended a pH range of 8.5 to 9.5 for protein based on legumes pea plant (Fedorovich, et al.,2009). Hence neutralization is done until pH value of 8±0.5 is achieved and finally filtration is done to extract sesame surfactant solution. On the other hand, for hingot extraction, hydothermolysis is carried out by heating solution of hingot powder in water at particular concentration in magnetic stirrer at a specified temperature followed by filtration. Previous studies have showed that the soaking time could be reduced through an accelerated process by heating the water at different temperatures (i.e. 70 °C to 90 °C) [Siva et al, 2016] without affecting the foaming ability. The surfactant solution is analysed with FTIR (Model: PerkinElmer, Spectrum two) to verify the extracted substances with reported literature. Surfactant viscosity is determined with Anton Par Rheometer with speeds ranging from 150 rpm to 200 rpm at a constant temperature of 25°C. The speed range was adopted on the basis of the manufacturer's recommendation and methodology as reported by various researchers in the literature (Sahu and Gandhi, 2021; Siva et al., 2015). Based on the experimental outcomes of present study it was observed that within the range of 150 to 200 rpm (shear rate- 194/s to 258/s) there was not much variation in the viscosity of surfactant solution. However, it is reported in literature that when the shear rate is reduced below 100/s the change in shear rate tends to affect the viscosity to a great extent (Raj et al., 2022). For foam production, a field-based model of foam generator supplied by Green Stone company is used, as shown in Figure 4-2 and the foam is generated based on the compressed air process. Optical microscopy coupled with Image J software is used to study the foam bubble microstructure. The initial foam density refers to the density of foam measured immediately after its generation. In the current study, foam stability is evaluated using the free drainage test as specified by Def-Standard 42-40. The drainage pan for the foam drainage system has a conical base and a nominal volume of 1.612 litres. A polymethyl methacrylate tube with an internal diameter of 12.7 mm and a length of 25 mm is fitted to the drainage pan's conical base. This pan is filled with foam, and at various time intervals, the volume of the solution drained is measured. OPC conforming to IS 269 is used to render foam cement paste.

Figure 4-1 (a) Hingot fruit (b) Sesame seed

Figure 4-2 Foam generator set up

4.3 Experiment Design, Model Formulation and Validity of Models

The optimization of surfactant extraction and foam production processes are carried out systematically based on response surface methodology (RSM) using Design Expert software. The parameters considered concerning the extraction process of sesame and hingot surfactants and foam production are surfactant concentration, temperature and duration of heating and FGP. However, for the sesame surfactant extraction process, adding to the above, sodium hydroxide concentration (alkali for hydrolysis) is also considered as an additional parameter. But, the ammonium chloride used for the neutralization process is not considered a variable input parameter for design, rather its dosage is chosen in such a way that pH of surfactant solution is maintained at 8±0.5 as recommended in earlier studies (Khwairakpam and Gandhi, 2021; Fedorovich et al., 2009).

Based on preliminary studies, surfactant concentration and FGP levels are fixed for the experimental design as shown in Table 4-1 and 4-2. For example, a surfactant solution with 2% surfactant concentration is prepared by adding 2 grams of extracted surfactant to 100 ml of distilled water. Different FGP levels are fixed for two surfactants based on the pressure required to maintain continuous foam generation. The performance of foam and surfactant is evaluated through measurement of the following responses viz., IFD, foam stability, bubble size and viscosity of surfactant solution. To derive the experiment treatments, Central composite design is adopted which includes 2n factorial design points, 2n axial design points, and centre point (repeated), where n is the number of factors. The centre point is repeated six times to provide information about reproducibility, and experiments are randomized to minimize unexplained variability in the actual responses due to extraneous factors. Various experimental treatments derived using Design expert is presented in Table 4-3 and 4-4. The response data obtained are analysed statistically using ANOVA and details of RSM models are presented in Table 4-5 and 4-6. The adequacy of response models is determined using model analysis, coefficient of determination (R^2) analysis, by examining the residuals for trends in diagnostic plots and by comparing the experimental data with values predicted by response surface models (Montgomery, 2001). RSM derived for various responses are found to be statistically significant ($p < 0.05$) with most of the main effects of independent variables having significant effect on response (Table 4-7 to 4-10). Further the model adequacy is checked based on R^2 and Adj R^2 values and comparison of model predicted values with actual experimental values. Analysis indicates that all the models have high R^2 and Adj R^2 values and predicted values are in well agreement with the actual values highlighting the model adequacy (Table 4-11 and 4-12). Further, multiple optimizations of RSM models are carried out to predict optimum levels of independent variables. Also, the check of the normality assumption may be made by constructing a normal probability plot of the residuals, as in Figure 4.3 to 4.8. Since the residuals plot approximately along a straight line, hence the normality assumption is satisfied. Figure 4.3 to 4.8 present a plot of residuals versus the predicted response. The residuals randomly scatter on the display suggesting that the variance of the original observations is constant for all the values of y.

Table 4-1 Factors and factor levels for sesame surfactant

Notation	**Factor**	**Low level**	**High level**
C	Surfactant concentration (%)	2	10
H	Sodium hydroxide (%)	2	10
T	Heating temperature (°C)	50	100
D	Heating duration (Hrs.)	3	10
GP	Foam generation pressure (kPa)	300	500

Table 4-2 Factors and factor levels for hingot surfactant

Notation	**Factor**	**Low level**	**High level**
SC	Surfactant concentration (%)	2	10
HT	Heating temperature (°C)	50	100
HD	Heating duration (Hrs.)	3	10
FGP	Foam generation pressure (kPa)	300	550

Table 4-3 Matrix of central composite design (CCD) for sesame surfactant

Run	**Sodium hydroxide concentration (%)**	**Sesame concentration (%)**	**Heating temperature (ºC)**	**Heating Duration (Hours)**	**Foam generation pressure (kPa)**
1	7.7	7.7	64	5.0	442
2	6.0	10.0	75	6.5	400
3	7.7	4.3	86	8.0	358
4	4.3	4.3	86	5.0	358
5	2.0	6.0	75	6.5	400
6	7.7	4.3	64	8.0	442
7	4.3	7.7	64	5.0	358
8	4.3	7.7	86	5.0	358
9	4.3	7.7	64	5.0	442
10	4.3	4.3	64	8.0	358
11	7.7	4.3	64	5.0	358
12	6.0	6.0	100	6.5	400
13	6.0(C)	6.0	75	6.5	400
14	6.0(C)	6.0	75	6.5	400
15	7.7	7.7	86	8.0	358
16	4.3	7.7	64	8.0	442
17	7.7	7.7	86	5.0	358
18	7.7	4.3	86	8.0	442
19	6.0	6.0	75	6.5	500
20	6.0	2.0	75	6.5	400
21	10.0	6.0	75	6.5	400
22	4.3	4.3	64	5.0	358
23	6.0(C)	6.0	75	6.5	400
24	7.7	4.3	86	5.0	442
25	4.3	7.7	86	8.0	358
26	7.7	4.3	86	5.0	358
27	6.0	6.0	50	6.5	400
28	4.3	4.3	64	5.0	442

Run	**Sodium hydroxide concentration (%)**	**Sesame concentration (%)**	**Heating temperature (ºC)**	**Heating Duration (Hours)**	**Foam generation pressure (kPa)**
29	7.7	4.3	64	8.0	358
30	6.0(C)	6.0	75	6.5	400
31	6.0	6.0	75	10.0	400
32	4.3	4.3	64	8.0	442
33	7.7	7.7	64	8.0	442
34	7.7	7.7	86	5.0	442
35	7.7	7.7	64	8.0	358
36	6.0(C)	6.0	75	6.5	400
37	4.3	7.7	86	5.0	442
38	6.0	6.0	75	6.5	400
39	7.7	4.3	64	5.0	442
40	7.7	7.7	86	8.0	442
41	4.3	4.3	86	5.0	442
42	4.3	4.3	86	8.0	358
43	4.3	4.3	86	8.0	442
44	6.0(C)	6.0	75	6.5	400
45	4.3	7.7	86	8.0	442
46	6.0	6.0	75	6.5	400
47	6.0	6.0	75	3.0	400
48	7.7	7.7	64	5.0	358
49	4.3	7.7	64	8.0	358
50	6.0	6.0	75	6.5	300

C: Centre point

Table 4-4 Matrix of central composite design (CCD) for hingot surfactant

Run	Hingot concentration (%)	Heating temperature (°C)	Heating Duration (Hours)	Foam generation pressure (kPa)
1	8	88	4.75	362.5
2	8	88	4.75	487.5
3	6	50	6.5	425
4	4	88	8.25	487.5
5	6	75	10	425
6	6(C)	75	6.5	425
7	8	63	8.25	487.5
8	6 (C)	75	6.5	425
9	4	88	4.75	487.5
10	6(C)	75	6.5	425
11	6(C)	75	6.5	425
12	6	100	6.5	425
13	4	88	4.75	362.5
14	6	75	6.5	550
15	4	88	8.25	362.5
16	2	75	6.5	425
17	8	63	4.75	362.5
18	6(C)	75	6.5	425
19	4	63	4.75	362.5
20	8	63	8.25	362.5
21	8	63	4.75	487.5
22	6(C)	75	6.5	425
23	4	63	8.25	487.5
24	4	63	4.75	487.5
25	8	88	8.25	362.5
26	6	75	6.5	300
27	6	75	3	425
28	10	75	6.5	425
29	4	63	8.25	362.5
30	8	88	8.25	487.5

C: Centre point

Table 4-5 RSM for different properties of foam and sesame surfactant solution

Properties	Response surface models
IFD (kg/m^3)	276.741 + -13.4775 × SC + -10.5709 ×SH + 2.2969 ×HT + -7.78952 ×HD + -0.559933 ×FGP + -0.342505 ×SC ×SH + -0.0053033 ×SC ×HT + 0.239911 ×SC ×HD + -0.0145841 ×SC ×FGP + -0.0901561 ×SH ×HT + -0.315673 ×SH ×HD + -0.00220971 ×SH ×FGP + -0.0828325 ×HT ×HD + -0.000353553 ×HT ×FGP + 0.0207081 ×HD ×FGP + 1.54782 ×SC^2 + 1.76657 ×SH^2 + -0.00997577 ×HT^2 + 0.144093 ×HD^2 + 0.000476514 ×FGP^2
Foam drainage 5th minute (%)	230.068 + -9.50904 ×SC + -6.34107 ×SH + 0.0246135 ×HT + -4.25917 ×HD + -0.572446 ×FGP + -0.220971 ×SC ×SH + -0.00353553 ×SC ×HT + 0.151523 ×SC ×HD + -0.0106066 ×SC ×FGP + -0.0742462 ×SH ×HT + -0.202031 ×SH ×HD + 0.00353553 ×SH ×FGP + -0.0606092 ×HT ×HD + -0.000707107 ×HT ×FGP + 0.00404061 ×HD ×FGP + 1.12339 ×SC^2 + 1.06089 ×SH^2 + 0.00555876 ×HT^2 + 0.487692 ×HD^2 + 0.000697423 ×FGP^2
Foam drainage 10th minute (%)	424.866 + -12.5461 ×SC + -7.07541 ×SH + -0.924021 ×HT + -13.3505 ×HD + -1.08593 ×FGP + -0.143631 ×SC ×SH + 0.00883883 ×SC ×HT + 0.416688 ×SC ×HD + -0.0145841 ×SC ×FGP + -0.0477297 ×SH ×HT + -0.492449 ×SH ×HD + -0.00574524 ×SH ×FGP + -0.026264 ×HT ×HD + 0.00176777 ×HT ×FGP + 0.0106066 ×HD ×FGP + 1.22187 ×SC^2 + 1.3625 ×SH^2 + 0.00207991 ×HT^2 + 0.718363 ×HD^2 + 0.00112999 ×FGP^2
Foam drainage 15th minute (%)	405.509 + -9.96111 ×SC + -10.8779 ×SH + -1.43262 ×HT + -12.1144 ×HD + -0.821292 ×FGP + -0.331456 ×SC ×SH + 0.00707107 ×SC ×HT + 0.3283 ×SC ×HD + -0.0203293 ×SC ×FGP + -0.0424264 ×SH ×HT + -0.429315 ×SH ×HD + -0.00265165 ×SH ×FGP + -0.0646498 ×HT ×HD + 0.00113137 ×HT ×FGP + 0.00909137 ×HD ×FGP + 1.3423 ×SC^2 + 1.56105 ×SH^2 + 0.00876284 ×HT^2 + 0.896063 ×HD^2 + 0.000897677 ×FGP^2
Foam bubble (µm)	954.595 + -35.9395 ×SC + -13.564 ×SH + -0.593161 ×HT + 8.53707 ×HD + -2.80169 ×FGP + -0.331456 ×SC ×SH + 0.0671751 ×SC ×HT + 0.151523 ×SC ×HD + 0.0344715 ×SC ×FGP + -0.0106066 ×SH ×HT + 0.101015 ×SH ×HD + 0.0256326 ×SH ×FGP + 0.00808122 ×HT ×HD + -0.00381838 ×HT ×FGP + -0.0545482 ×HD ×FGP + 0.936488 ×SC^2 + 0.217738 ×SH^2 + 0.00877409 ×HT^2 + 0.52929 ×HD^2 + 0.00339838 ×FGP^2

Surfactant concentration (SC), Heating temperature (HT), heating duration (HD) and foam generator pressure (FGP), Sodium hydroxide (SH)

Table 4-6 RSM for different properties of foam and hingot surfactant

Properties	Response surface models
IFD (kg/m^3)	$674.248 + -15.4357 \times SC + -2.73325 \times HT + -18.1244 \times HD + -1.53708 \times FGP + -0.0245 \times SC \times HT + -0.182143 \times SC \times HD + 0.0001 \times SC \times FGP + 0.0405714 \times HT \times HD + -0.001296 \times HT \times FGP + 0.0104 \times HD \times FGP + 1.29115 \times SC^2 + 0.0166533 \times HT^2 + 0.645578 \times HD^2 + 0.00170613 \times FGP^2$
Foam drainage 5th minute (%)	$264.5 + -14.3092 \times SC + -1.61164 \times HT + -1.83513 \times HD + -0.425143 \times FGP + 0.012325 \times SC \times HT + 0.24625 \times SC \times HD + -0.004655 \times SC \times FGP + -0.0112857 \times HT \times HD + 0.0015576 \times HT \times FGP + 0.00240571 \times HD \times FGP + 1.02586 \times SC^2 + 0.004966 \times HT^2 + -0.0866327 \times HD^2 + 0.00029656 \times FGP^2$
Foam drainage 10th minute (%)	$363.779 + -16.3399 \times SC + -2.42705 \times HT + -3.53946 \times HD + -0.636724 \times FGP + 0.02 \times SC \times HT + 0.178571 \times SC \times HD + -0.006 \times SC \times FGP + -0.0114286 \times HT \times HD + 0.00176 \times HT \times FGP + 0.00228571 \times HD \times FGP + 1.21354 \times SC^2 + 0.00946667 \times HT^2 + 0.0748299 \times HD^2 + 0.000538667 \times FGP^2$
Foam drainage 15th minute (%)	$195.496 + -9.29762 \times SC + -1.14105 \times HT + 4.14558 \times HD + -0.195771 \times FGP + 0.015 \times SC \times HT + 0.0714286 \times SC \times HD + -0.02 \times SC \times FGP + -0.0114286 \times HT \times HD + 0.00224 \times HT \times FGP + -0.00114286 \times HD \times FGP + 1.17708 \times SC^2 + -0.000266667 \times HT^2 + -0.340136 \times HD^2 + 8.53333 \times 10^{-5} \times FGP^2$
Foam bubble (µm)	$63.6258 + -2.65506 \times SC + 4.84233 \times HT + -11.3534 \times HD + 0.230829 \times FGP + -0.0475 \times SC \times HT + 0.160714 \times SC \times HD + -0.0055 \times SC \times FGP + -0.06 \times HT \times HD + -0.00488 \times HT \times FGP + -0.00514286 \times HD \times FGP + -0.101563 \times SC^2 + -0.0186 \times HT^2 + 1.09184 \times HD^2 + 5.6 \times 10^{-5} \times FGP^2$
Viscosity (mPa-sec)	$-0.654575 + -0.0233127 \times SC + 0.0580023 \times HT + -0.158404 \times HD + 0.000707107 \times SC \times HT + 0.0151523 \times SC \times HD + -0.00153543 \times HT \times HD + 0.0023044 \times SC^2 + -0.000285007 \times HT^2 + 0.0197445 \times HD^2$

Surfactant concentration (SC), Heating temperature (HT), heating duration (HD) and foam generator pressure (FGP)

Table 4-7 ANOVA and regression coefficients of response surface models fitted (Main effects and Quadratic effects -Sesame)

Variables		Main effects					Quadratic effects				
		x_1	**x_2**	**x_3**	**x_4**	**x_5**	**x_1^2**	**x_2^2**	**x_3^2**	**x_4^2**	**x_5^2**
IFD (kg/m^3)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.0031	0.3693	0.02	< 0.0001
	F- value	50.0724	150.3213	266.5891	345.7569	23.81524	163.7082	10.3762	0.831665	6.06094	213.2511
Foam drainage 5th minute (%)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.0207	< 0.0001	0.018	0.0002	< 0.0001	< 0.0001
	F- value	28.2863	116.7146	37.3372	161.0052	5.991639	168.3796	6.29079	18.60169	25.3501	150.1651
Foam drainage 10th minute (%)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.0053	< 0.0001	0.4895	< 0.0001	< 0.0001	< 0.0001
	F- value	29.8317	76.28117	75.34623	134.0137	9.072021	110.8088	0.48992	22.45137	37.0199	137.7825
Foam drainage 15th minute (%)	p- value	0.0002	< 0.0001	< 0.0001	< 0.0001	0.0007	< 0.0001	0.0435	0.0002	0.0017	< 0.0001
	F- value	17.9773	36.35652	41.85125	61.74413	14.2788	68.52045	4.45586	17.89911	11.9707	92.67334
Foam bubble (µm)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.2174	0.0567	0.026	< 0.0001
	F- value	437.27	61.41	129.62	762.35	73.79	29.41	3.94	5.51	151.28	1.59
Viscosity (mPa-sec)	p- value	< 0.0001	< 0.0001	< 0.0001	-	1	< 0.0001	< 0.0001	< 0.0001	-	< 0.0001
	F- value	10628.9	697.5296	2196.062	-	0	765.44	346.28	91.22	-	310.57

x_1- surfactant concentration; x_2- temperature; x_3- duration; x_4- FGP; x_5 – NaOH concentration

Table 4-8 ANOVA and regression coefficients of response surface models fitted (Interaction effect -Sesame)

Variables		**Interaction effect**									
		x_1x_2	**x_1x_3**	**x_1x_4**	**x_1x_5**	**x_2x_3**	**x_2x_4**	**x_2x_5**	**x_3x_4**	**x_3x_5**	**x_4x_5**
IFD (kg/m^3)	p- value	0.8367	0.1982	0.0297	0.0402	0.0081	0.7314	0.0014	0.01	0.0938	0.7314
	F- value	0.043231	1.73406	5.231	4.61615	8.07467	0.1201	12.4939	8.07	3.002182	0.1201
Foam drainage	p- value	0.8478	0.2547	0.0273	0.0626	0.007	0.3408	0.0003	0.44	0.1321	0.4448
5^{th} minute (%)	F- value	0.037516	1.35058	5.40231	3.75161	8.44111	0.9379	16.5446	0.6	2.401028	0.6003
Foam drainage	p- value	0.7206	0.0239	0.0239	0.3555	0.3555	0.0813	0.0609	0.14	0.0086	0.3555
10^{th} minute (%)	F- value	0.130434	5.68171	5.68171	0.88174	0.88174	3.2609	3.80346	2.3	7.935616	0.8817
Foam drainage	p- value	0.8376	0.1893	0.0242	0.1317	0.1088	0.4148	0.2246	0.36	0.0893	0.7586
15^{th} minute (%)	F- value	0.042773	1.80716	5.65674	2.40598	2.73748	0.6844	1.53983	0.87	3.090353	0.0962
Foam bubble	p- value	0.0753	0.5647	0.0007	0.156	0.8474	0.0138	0.085	< 0.0001	0.7005	0.0087
(µm)	F- value	3.4	0.34	14.34	2.12	0.038	6.87	0.7729	27.5	0.15	7.93
Viscosity	p- value	< 0.0001	0.0009		< 0.0001	< 0.0001		< 0.0001		< 0.0001	
(mPa-sec)	F- value	351.37	36.39		890.11	351.37		351.37		1345.61	

x_1- surfactant concentration; x_2- temperature; x_3- duration; x_4- FGP; x_5 – NaOH concentration

Table 4-9 ANOVA and regression coefficients of response surface models fitted (Main effects and Quadratic effects -Hingot)

Variables		**Main effects**				**Quadratic effects**			
		x_1	**x_2**	**x_3**	**x_4**	**x_1^2**	**x_2^2**	**x_3^2**	**x_4^2**
IFD (kg/m^3)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.004	0.0214	< 0.0001
	F- value	50.41	103.4	51.13	77.46	45.03	11.43	6.6	74.98
Foam drainage 5th minute (%)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.006	0.2951	0.0003
	F- value	123.45	95.31	76.65	268.53	281.6	10.07	1.18	22.44
Foam drainage 10th minute (%)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.001	0.5378	< 0.0001
	F- value	73.62	47.12	38.7	119.68	178.43	16.57	0.4	33.53
Foam drainage 15th minute (%)	p- value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	0.912	0.0134	0.3843
	F- value	109.81	44.978	27.45242	168.843	160.247	0.013	7.84355	0.80318
Foam bubble (µm)	p- value	< 0.0001	< 0.0001	<0.0001	< 0.0001	0.6664	0.0067	0.0025	0.8161
	F- value	312.39	77.72	26.05	94.95	0.19	9.89	13.1	0.056
Viscosity (mPa-sec)	p- value	< 0.0001	< 0.0001	< 0.0001	-	0.5395	0.012	0.0019	-
	F- value	309.34	45.0189	53.4314	-	0.4	9.42	17.37	-

x_1- surfactant concentration; x_2- temperature; x_3- duration; x_4- FGP

Table 4-10ANOVA and regression coefficients of response surface models fitted (Interaction effect- Hingot)

Variables		**Interaction effect**					
		x_1x_2	x_1x_3	x_1x_4	x_2x_3	x_2x_4	x_3x_4
IFD (kg/m^3)	p- value	0.5524	0.5365	0.9903	0.3924	0.331	0.2767
	F- value	0.37	0.4	0.0001	0.78	1.01	1.27
Foam drainage 5th minute (%)	p- value	0.3511	0.0167	0.0892	0.4526	0.0017	0.424
	F- value	0.93	7.25	3.3	0.59	14.45	0.68
Foam drainage 10th minute (%)	p- value	0.31	0.2087	0.1358	0.607	0.0112	0.607
	F- value	1.1	1.73	2.48	0.28	8.35	0.28
Foam drainage 15th minute (%)	p- value	0.4532	0.6152	0.0001	0.6152	0.0027	0.8009
	F- value	0.5929	0.2635	26.354	0.2635	12.9136	0.0658
Foam bubble (µm)	p- value	0.3419	0.6486	0.5782	0.2951	0.0066	0.6486
	F- value	0.96	0.22	0.32	1.18	9.93	0.22
Viscosity (mPa-sec)	p- value	0.3853	0.02	-	0.1152689	-	-
	F- value	0.82	7.42	-	2.98	-	-

x_1- surfactant concentration; x_2- temperature; x_3- duration; x_4- FGP

Table 4-11 R^2, probability value and F values for the final models of sesame surfactant solution

Response variable	**R^2**	**R^2 adj**	**Regression p value**	**F -value**
IFD (kg/m^3)	0.9775	0.9619	<0.0001	62.93
Foam drainage 5th minute (%)	0.9590	0.9306	<0.0001	33.87
Foam drainage10th minute (%)	0.9537	0.9218	<0.0001	29.90
Foam drainage 15th minute (%)	0.9214	0.0.8672	<0.0001	17
Foam bubble (µm)	0.9832	0.9716	<0.0001	84.7
Viscosity (mPa-sec)	0.9952	0.9839	<0.0001	88.12

Table 4-12 R^2, probability value and F values for the final models of hingot surfactant

Response variable	**R^2**	**R^2 adj**	**Regression p value**	**F -value**
IFD (kg/m^3)	0.9633	0.9291	<0.0001	28.16
Foam drainage 5th minute	0.9835	0.9681	<0.0001	63.85
Foam drainage 10th minute (%)	0.9705	0.9430	<0.0001	35.29
Foam drainage 15th minute	0.9746	0.9509	<0.0001	41.10
Foam bubble (µm)	0.9735	0.9488	<0.0001	39.36
Viscosity (mPa-sec)	0.9574	0.9382	<0.0001	49.92

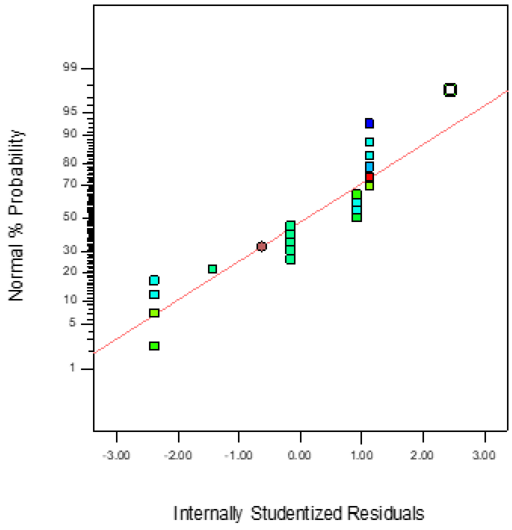


Figure 4-3 (a) Normality Probabilty plot (sesame)

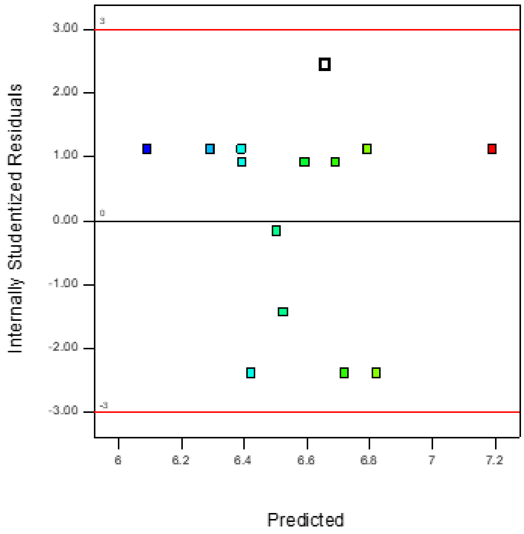


Figure 4-3 (b) Residual plot (sesame)

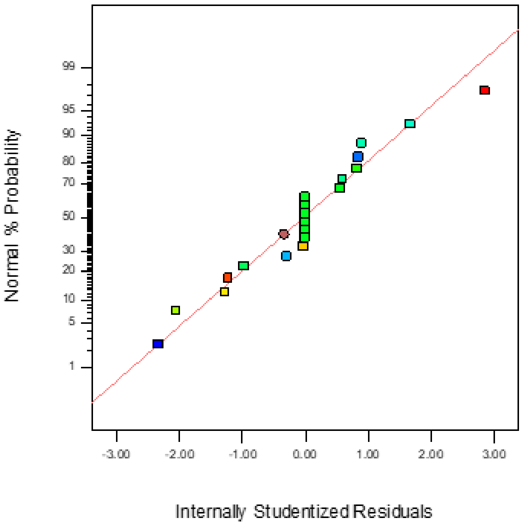


Figure 4-3 (c) Normality Probabilty plot (hingot)

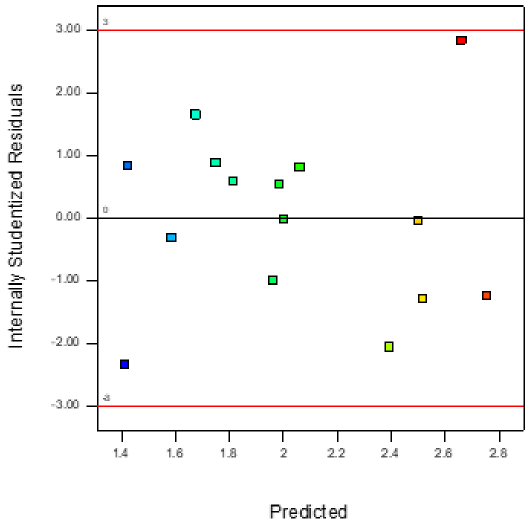


Figure 4-3 (d) Residual plot (hingot)

Figure 4-3 Diagnostic plots for viscosity of surfactant solution

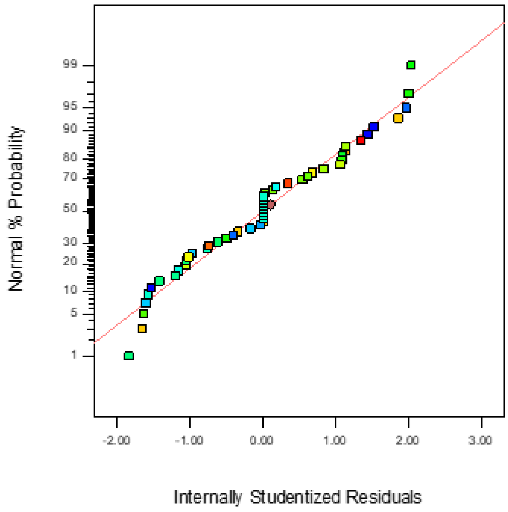


Figure 4-4 (a) Normality Probabilty plot (sesame)

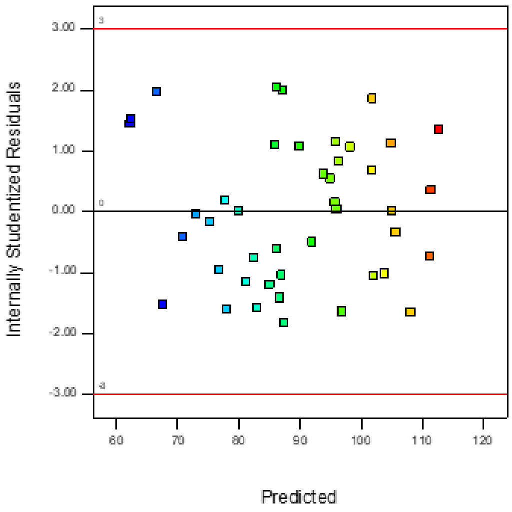


Figure 4-4 (b) Residual plot (sesame)

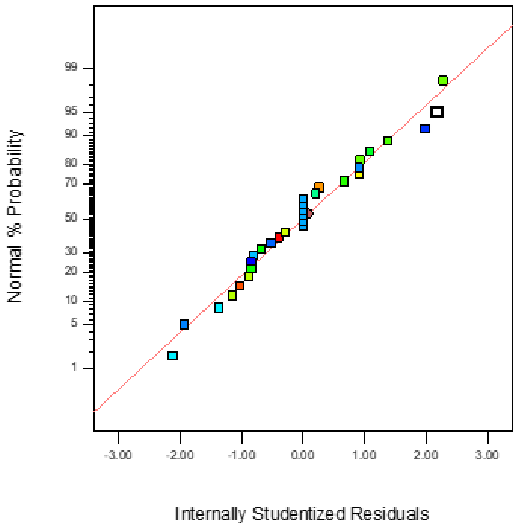


Figure 4-4 (c) Normality Probabilty plot (hingot)

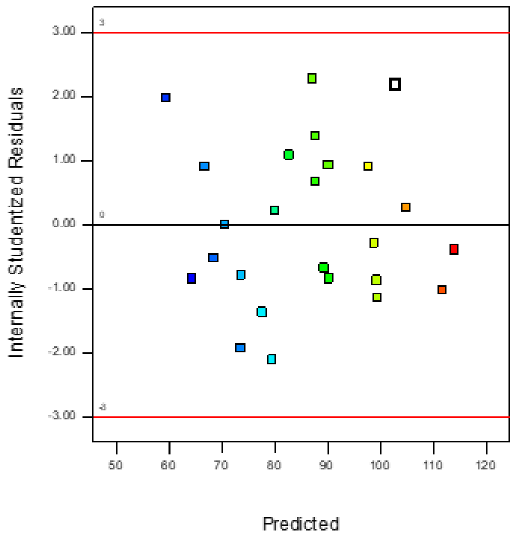
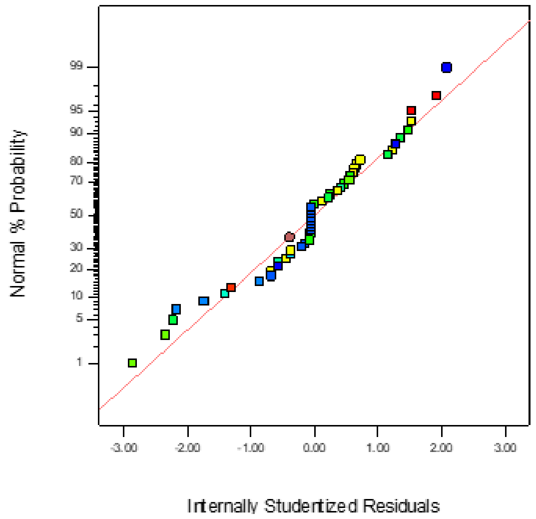


Figure 4-4 (d) Residual plot (hingot)

Figure 4-4 Diagnostic plots for IFD



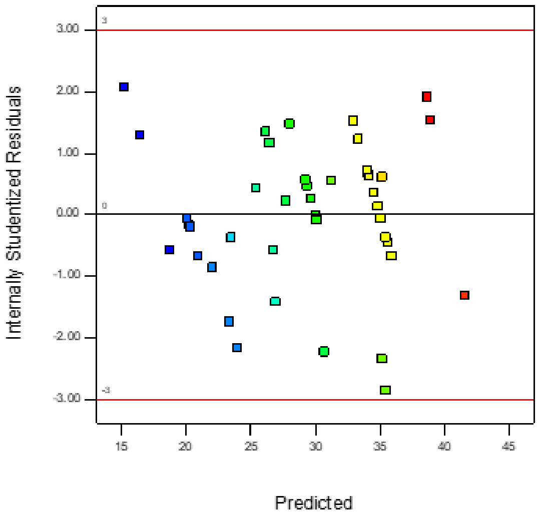
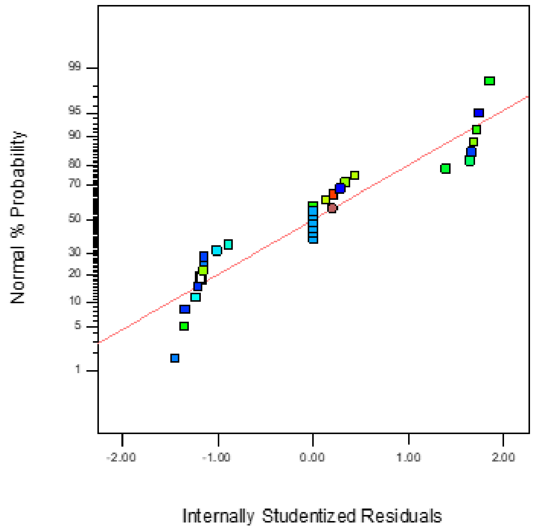


Figure 4-5 (a) Normality Probabilty plot (sesame)

Figure 4-5 (b) Residual plot (sesame)



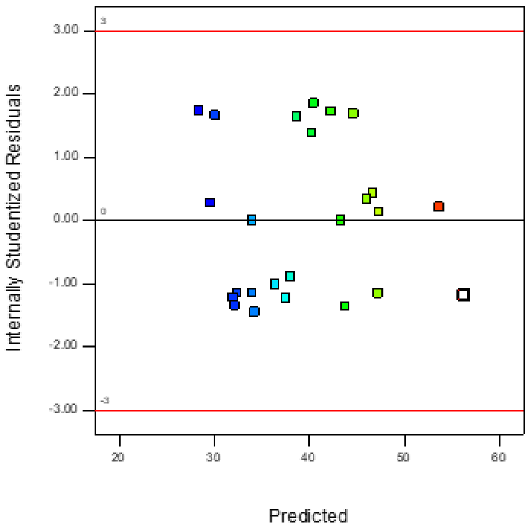
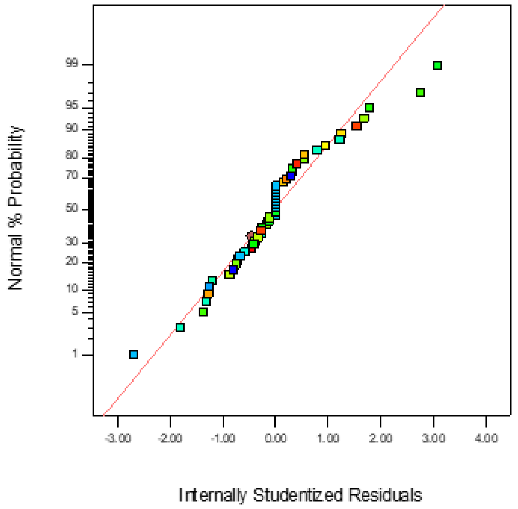


Figure 4-5 (c) Normality Probabilty plot (hingot)

Figure 4-5 (d) Residual plot (hingot)

Figure 4-5 Diagnostic plots for solution drained at 5th minute



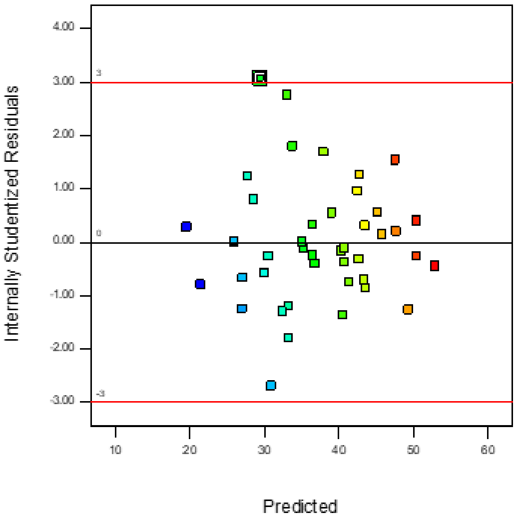
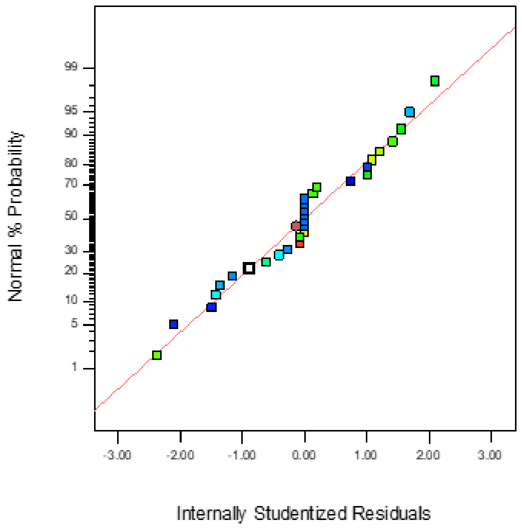


Figure 4-6 (a) Normality Probabilty plot (sesame)

Figure 4-6 (b) Residual plot (sesame)



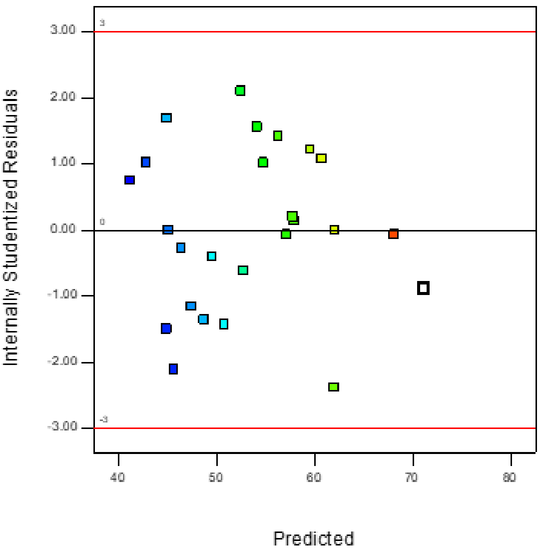


Figure 4-6 (c) Normality Probabilty plot (hingot)

Figure 4-6 (d) Residual plot (hingot)

Figure 4-6 Diagnostic plots for solution drained at 10th minute

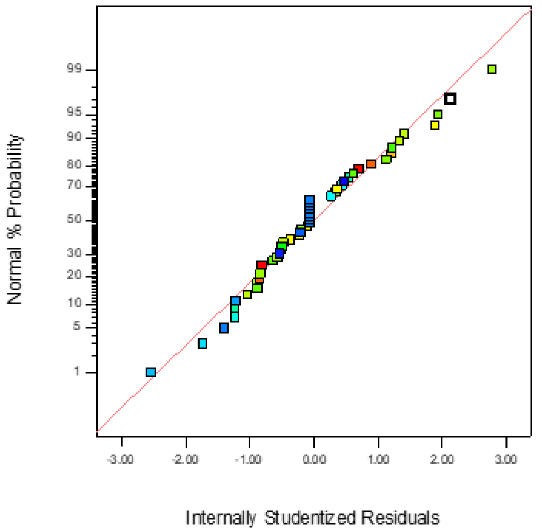


Figure 4-7 (a) Normality Probabilty plot (sesame)

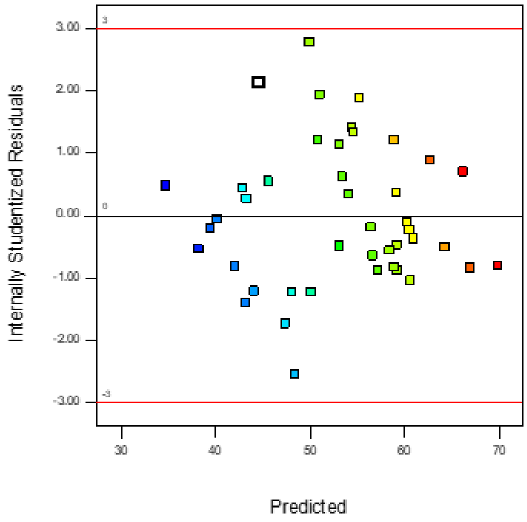


Figure 4-7 (b) Residual plot (sesame)

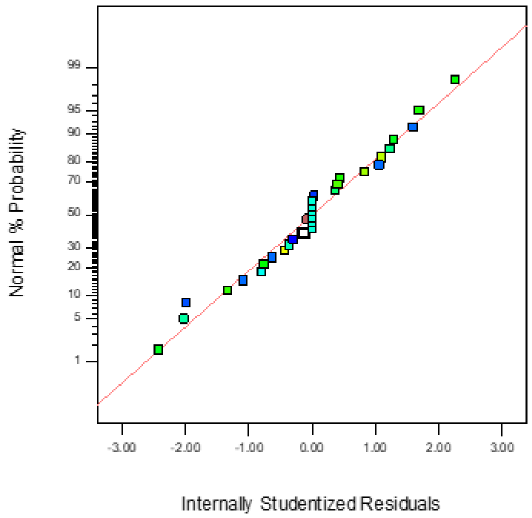


Figure 4-7 (c) Normality Probabilty plot (hingot)

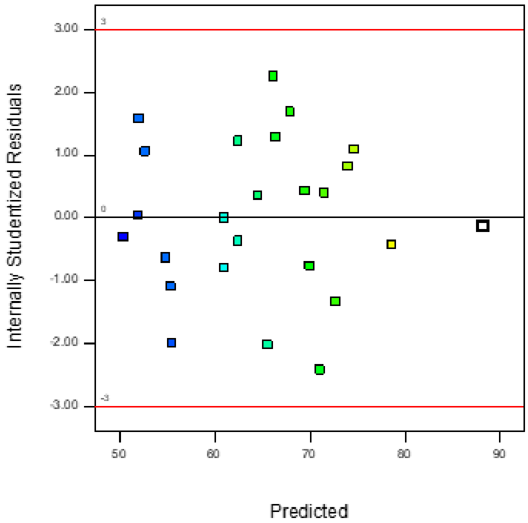
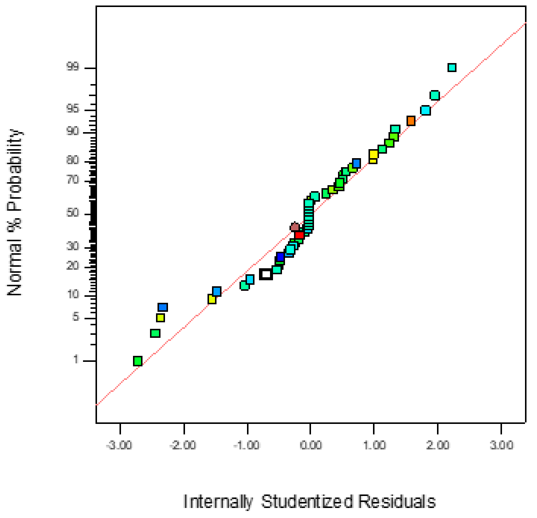


Figure 4-7 (d) Residual plot (hingot)

Figure 4-7 Diagnostic plots for solution drained at 15th minute



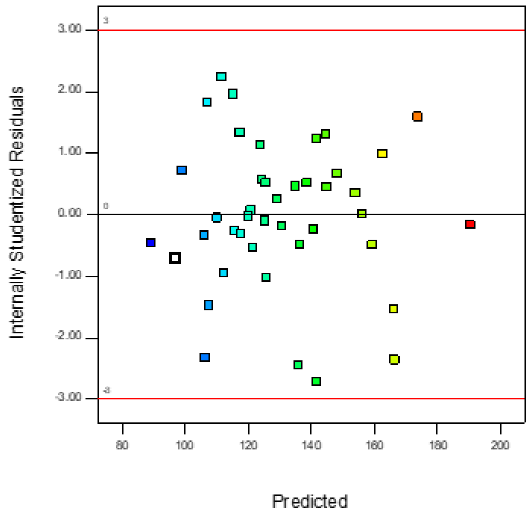
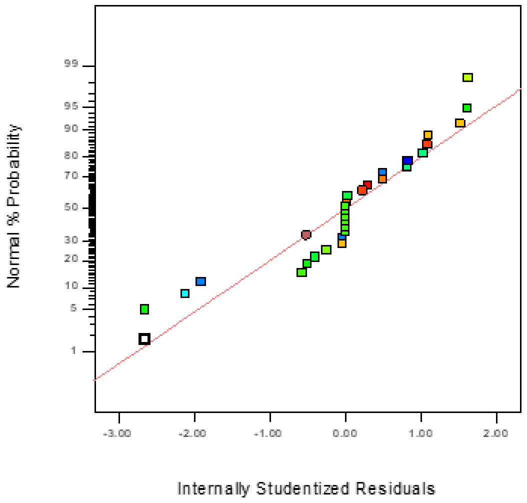


Figure 4-8 (a) Normality Probabilty plot (sesame)

Figure 4-8 (b) Residual plot (sesame)



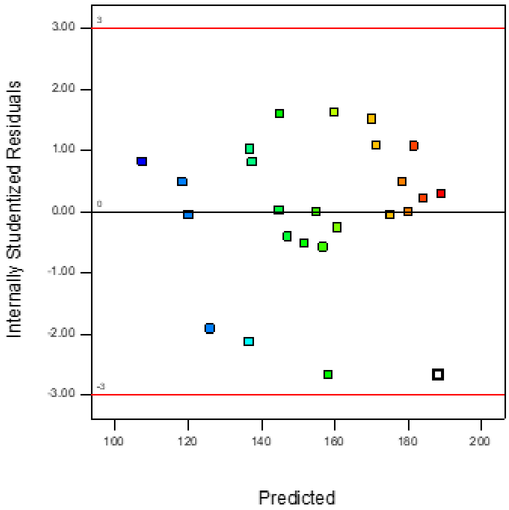


Figure 4-8 (c) Normality Probabilty plot (hingot)

Figure 4-8 (d) Residual plot (hingot)

Figure 4-8 Diagnostic plots for bubble size

4.4 Results and Discussion

4.4.1 FTIR analysis of surfactants

Infrared absorption spectrum is derived for both the surfactants (Figure 4-9) and compared with available literature. A liquid sample is prepared for IR analysis by placing a drop of liquid on a highly polished salt plate (such as NaCl, AgCl, or KBr), laying a second plate on top of the first plate, and clamping the plates together. FTIR transmittance spectrum of saponin surfactant shows that the hydroxyl group (-OH stretching) peak is at 3281.04 cm^{-1}. This observation is consistent with literature reported values of 3390 cm^{-1} (Yekeen et al., 2020), 3455 cm^{-1} (Samal et al., 2017), 3434 cm^{-1} (Bajad and Pardeshi, 2016). Carbon-hydrogen (C-H) absorption is observed at 2922.99 cm^{-1} while literature values are reported at 2939.60 cm-1, 2931 cm^{-1} (Yekeen et al., 2020). Similarly, Carbonyl (C=O) absorbance is noted at 1633.96 cm^{-1}, while earlier researchers have reported 1622.52 cm^{-1}, 1724 cm^{-1} (Yekeen et al., 2020). On a similar note, the transmittance spectra of all the other functional groups can be obtained from Figure 4-9 for both the surfactants. The output of FTIR analysis of extracted saponin is comparable with values reported for different saponins in literature and this confirms that the surfactant extracted from hingot is rich in saponin. On a similar note, FTIR transmittance spectrum of sesame surfactant appears much similar to saponin based hingot surfactant. However, unlike saponin based, the characteristics bands of asymmetric and symmetric stretching (CH) are clearly evident at 2922.62 cm^{-1} and 2853.09 cm^{-1}respectively. Furthermore, other characteristic bands of Absorbance of Hydroxyl group and Carbonyl occurred at bands of 3284.17 cm^{-1} and 1742 cm^{-1} respectively. This is consistent with values reported in literature for sesame oil (Gómez-Contreras et al., 2021). It is to be noted that though chemicals such as sodium hydroxide and ammonium chloride are used for extraction of the sesame surfactant, the FTIR spectra of surfactant derived in this study matches fairly with FTIR output of virgin sesame oil reported in literature.

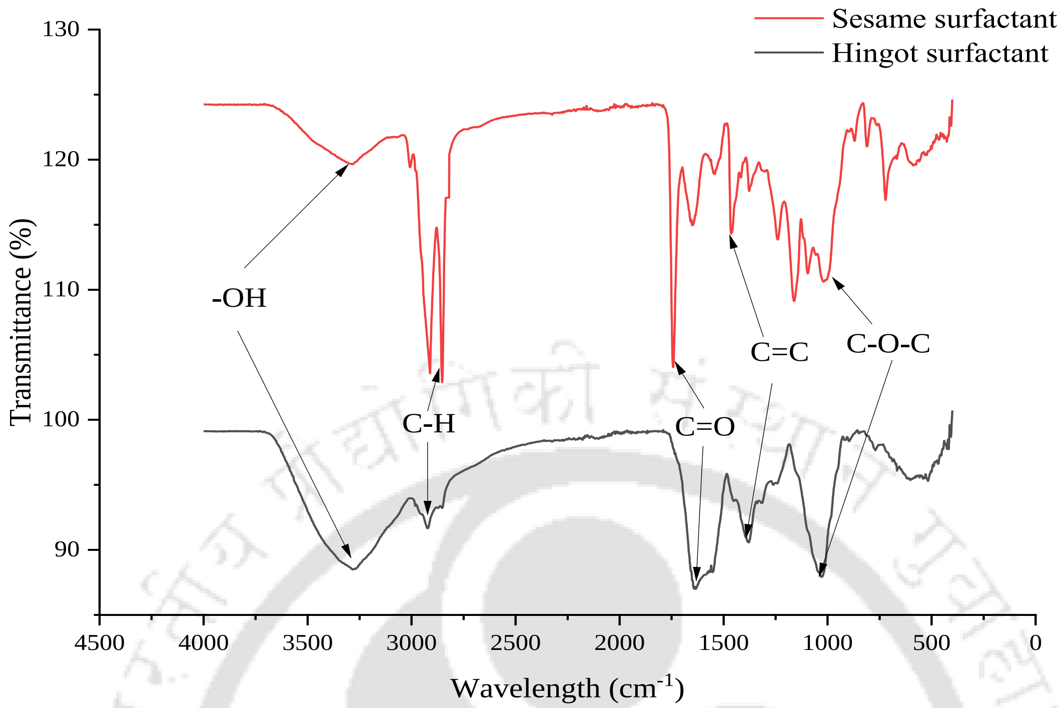


Figure 4-9 FTIR analysis chart and functional groups of extracted sesame and hingot surfactant

4.4.2 RSM models for viscosity of surfactant solution

RSM models for viscosity of surfactant solution are derived as shown in Table 4-5 and 4-6. To analyse the effects and interactions, ANOVA is done which indicates that all the main effects of independent variables (except NaOH concentration) and interaction effects have significant contribution to response with $p<0.05$ (Table 4-7 to 4-10). To study the variation of response, RSM plots are drawn for various combinations of independent variables as discussed below. Figure 4-10 shows the RSM plots for viscosity of hingot and sesame surfactant solution depicting the effect of variation of surfactant concentration and heating temperature of surfactant solution. The RSM plots of viscosity of both the surfactants are plotted for constant heating duration of 5 hours as mentioned in Figure 4-10. In addition, the viscosity plot for sesame surfactant corresponds to NaOH concentration of 5%. Figure 4-10 illustrates that when surfactant concentration increases, the viscosity of the surfactant solution increases. An increase of 89 % in viscosity is observed with an increase in hingot level from 2 to 10% at a constant heating temperature of 50^{o}C. As observed in previous investigations by other researchers, the above-mentioned increase in viscosity can be attributed to change in molecular arrangement with increase in surfactant concentration (Sharma and Sharma, 1991; Cornwell, 2018). Generally increase in surfactant concentration results in formation of micelles. The formation of micelles in a solution can increase its viscosity, or resistance to flow, because the micelles act as obstacles to the movement of other molecules in the solution. The size and shape of the micelles depend on the concentration of the surfactant in the solution. At low concentrations, the surfactant molecules are dispersed in the solution and do not form micelles. As the concentration of surfactant increases, more and more micelles form, and the size and number of these micelles can increase as well thus leading to increase in viscosity (Miller, 2007). Further, Cornwell substantiated that as the concentration is raised, the micelles elongate into rods that eventually stack to form a high-viscosity hexagonal phase. Experimental results indicate that though the sesame surfactant solution is highly viscous when compared to hingot solution, the variation in viscosity with increase in sesame surfactant concentration is relatively lesser than hingot.

As the degree of hydrolysis of protein-based as well as saponin based surfactants are very much dependent on the heating process of surfactant solution, it is essential to determine the optimal temperature and duration of the heating process for surfactant extraction. Considering the effect of heating temperature on viscosity of surfactant solution, it is observed from Figure 4-10, increase in heating temperature of hingot surfactant solution from 50 to 100^{o}C results in 45% increase in viscosity of surfactant solution. Enhancement of hydrolysis of saponin with increase in heating temperature of surfactant solution can be ascribed to increase in viscosity of solution. In other words, increase in temperature leads to accelerated extraction of the surfactant molecules from the surfactant sources. This in turn results in increase in surfactant molecules concentration subsequently enhancing viscosity.

However, for sesame surfactant, the effect of increase in heating temperature on viscosity of surfactant solution is not noteworthy. To study the effect of heating duration, RSM plots of viscosity depicting the effect of variation of surfactant concentration and heating duration at constant heating temperature of 90^{o}C and constant NaOH concentration of 5% are drawn in Figure 4-11. From the graph it is observed that increase in heating duration, the viscosity of the surfactant solution increases as a result. For hingot surfactant, when heating duration is increased from 3 to 10 hours, 29 % increase in viscosity is observed for surfactant concentration of 10%. However, for low hingot surfactant concentration, increase in heating duration has not much effect on viscosity indicating interaction between two factors. The above interaction between hingot surfactant concentration and heating duration is represented graphically in Figure 4-12. Further, in line with earlier discussion, heating temperature as well as heating duration have comparatively lesser effect when associated to surfactant concentration on viscosity of sesame surfactant solution.

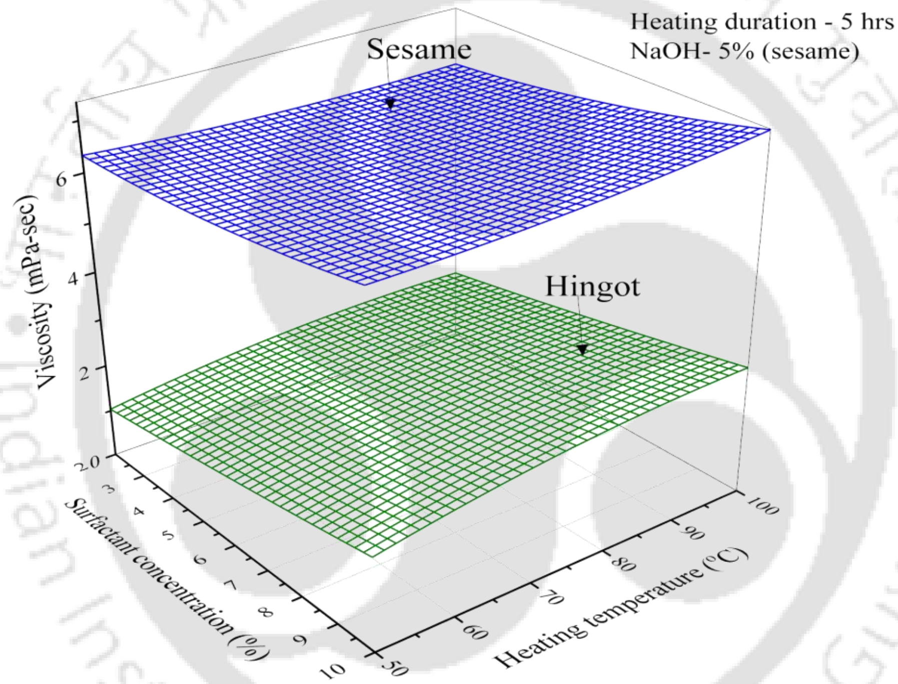


Figure 4-10 Variation of viscosity with surfactant concentration and heating temperature

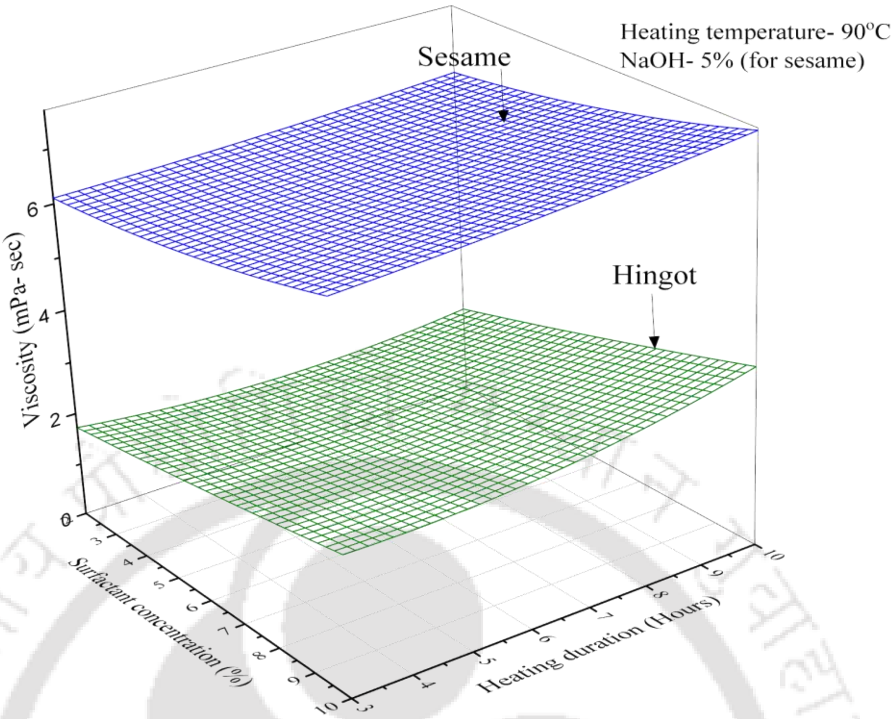


Figure 4-11 Variation of viscosity with surfactant concentration and heating duration

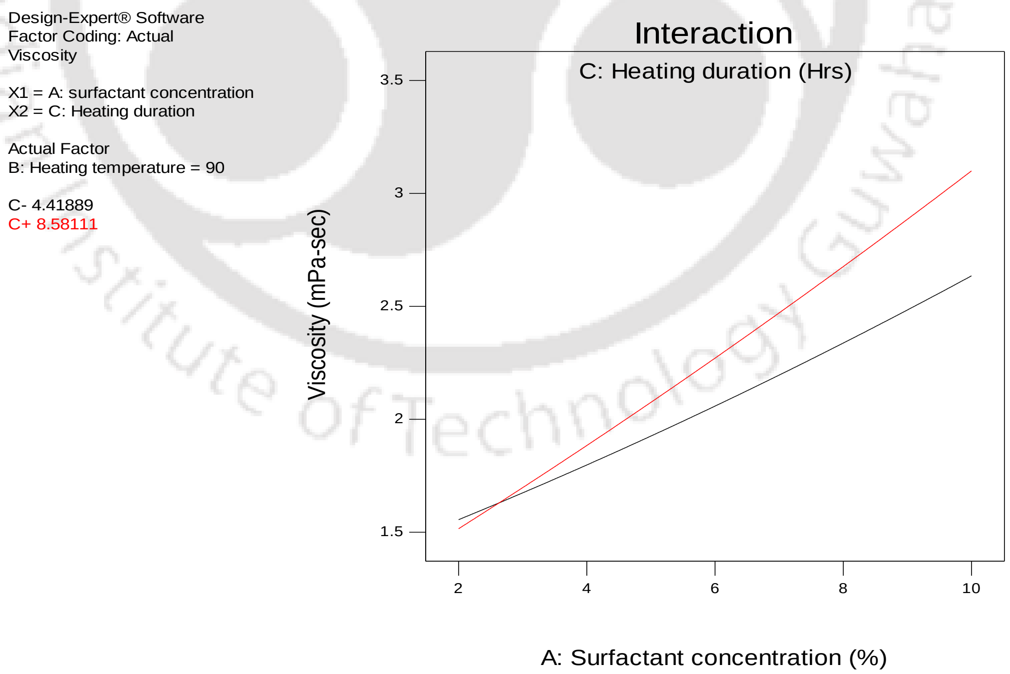


Figure 4-12 Interaction plot for surfactant concentration and heating duration (hingot surfactant)

Furthermore, RSM plot on the effect of variation of NaOH concentration for sesame surfactant indicates that viscosity of sesame surfactant solution is maximized when higher surfactant concentration and higher NaOH concentration is adopted (Figure 4-13). Also, the statistical analysis shows that the main effect of NaOH concentration on viscosity is insignificant, nevertheless the interaction effects are significant (Table 4-7 and 4-8). For instance, at constant NaOH concentration of 10%, when surfactant concentration is increased from 2 to 10%, 39% increase in viscosity is observed. But at low NaOH concentration of 2%, increase in surfactant concentration has no significant effect on viscosity indicating significant interaction between surfactant concentration and NaOH concentration with $p<0.05$. Hence, it is evident that right combination of surfactant concentration and NaOH concentration is needed for optimum hydrolysis of protein. Generally, larger F-value and smaller p-value of a model factor signify its respective significance to the response variable. Considering the main effects of all independent variables, surfactant concentration is found to have substantial effect on variation in viscosity when compared to other main effects for both the surfactants. The above observation is justified by higher F value of the main effect of surfactant concentration (Table 4-7 and 4-9).

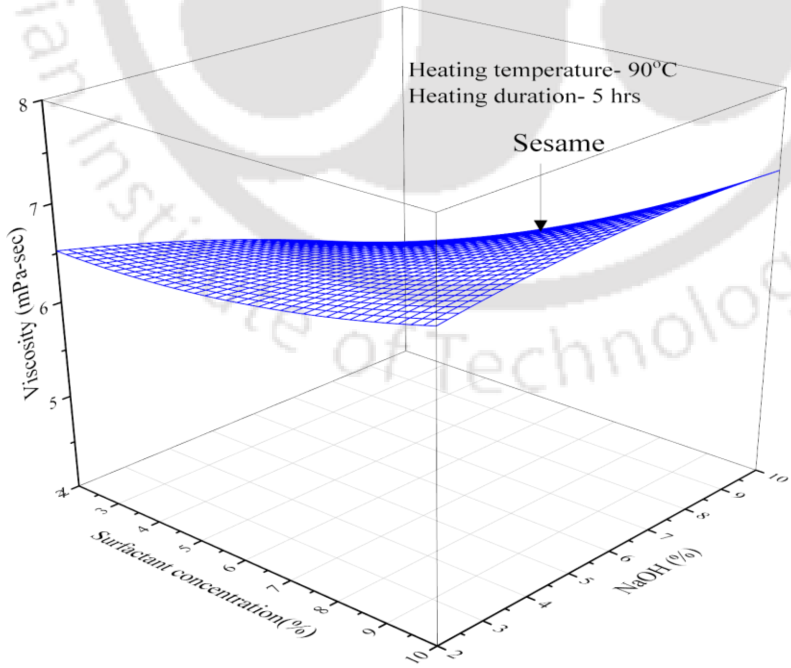


Figure 4-13 Variation of viscosity with surfactant concentration and NaOH concentration

4.4.3 RSM models for IFD

Statistical analysis of RSM plots of IFD, when compared to quadratic and interaction effects, all main effects of independent variables have a significant effect ($p<0.05$) (Table 4-7 to 4-10). For the systematic analysis, firstly, the effect of variation of surfactant concentration and FGP on IFD of produced foam for both the surfactants are scrutinized graphically (Figure 4-14). The RSM plot of IFD for both the surfactants are plotted at constant heating temperature of 90^{o}C and duration of 5 hours. For sesame seed surfactant, the variation of IFD is studied at constant NaOH concentration of 5%. It is observed from Figure 4-14 that at higher surfactant concentration for both the surfactants, IFD reduces drastically with increase in FGP. For case, a substantial reduction of 41% in IFD is observed with increase in FGP from 300 to 500 kPa for 10% sesame surfactant concentration. The above observations indicate that for compressed air method of foam production, FGP is a significant parameter as justified by its highest F ratio when compared to main effects of other independent variables particularly for sesame surfactant (Table 4-7 and 4-9). This observation supports the notion that a higher level of FGP is required for adequate air and liquid mixing to result in good quality foam (Sahu and Gandhi, 2021).

On further examination of convex RSM plots drawn for IFD (Figure 4-14), it is evident that, with increase in surfactant concentration (SC), the quality of foam increases. This is evident from the reduction in the liquid fraction of the foam (i.e reduction in initial foam density) with increase in SC up to 7%. The reduction in foam liquid fraction with increase in SC as observed in this study is consistent with literature (Osei-Bonsu et al., 2015). Hence up to SC of 7%, though viscosity increased with increase in SC, reduction in IFD is observed.

However, after a certain SC (say 7%), increase in IFD is observed with increase in concentration. This increase in IFD beyond inflection point of 7% can be ascribed to increased viscosity of solution at higher SC. Similar such observations were reported in previous studies (Sahu et al., 2021; Porter, 1994). As there is a wide variation in IFD from 124 to 75 kg/m^3, the behaviour of foam when SC is less than 7% is completely different from its behaviour above SC of 7%. When SC is less than 7%, as the foam is very wet with high liquid fraction, it is evident that IFD reduces with increase in viscosity. However, beyond SC of 7%, different correlation between viscosity and IFD is observed.

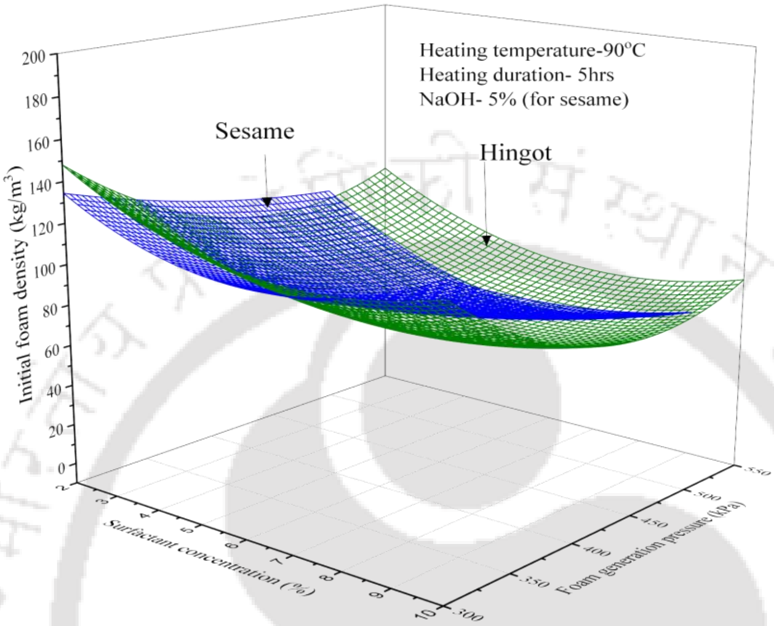


Figure 4-14 Variation of IFD with surfactant concentration and FGP

Further, to investigate the effect of variation in heating temperature, RSM plots of IFD are drawn for both the surfactants at constant heating duration of 5 hours, FGP of 500kPa and NaOH concentration level of 5% (Figure 4-16). In line with earlier discussion on viscosity, heating temperature has positive effect on improvement of quality of foam. In the case of both surfactants, increase in heating temperature of surfactant solution results in 11 to 38% reduction of IFD for the range of parameters studied. The above reduction in IFD and enhancement in quality of foam can be attributed to enhancement of hydrolysis of protein and saponin with increase in heating temperature of surfactant solution. Generally, heating can increase the diffusion rate, the convection of the solvent, thus increasing the extraction efficiency. As temperatures increase, saponins move more flexibly and mass transfer increases, thereby increasing the saponin content. However, when the saponin extraction process reaches the saturation state, further increase in temperature is no longer effective and only result in increase in cost (Nhat et al., 2019). It is also reported in literature initiation of degradation of saponin can happen at very high temperatures. Hence an optimum temperature has to be determined for extraction. On a similar note, considering the effect of heating duration, it is observed from Figure 4-17 that for both the surfactants with increase in heating duration of surfactant preparation solution, reduction in IFD (reduction of the liquid fraction of foam) is observed.

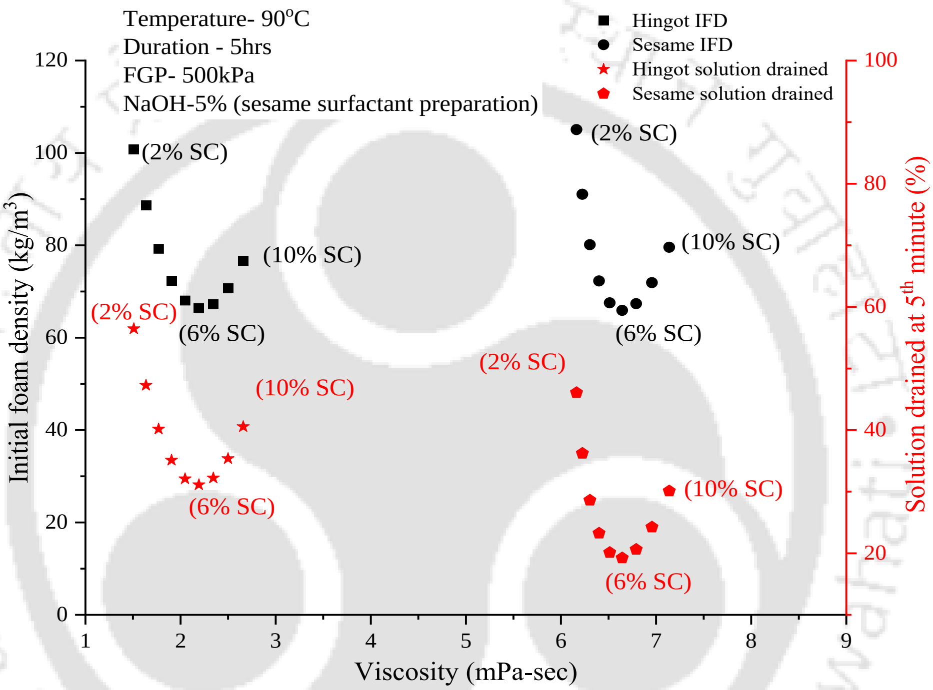


Figure 4-15 Relationship between viscosity, IFD and solution drained

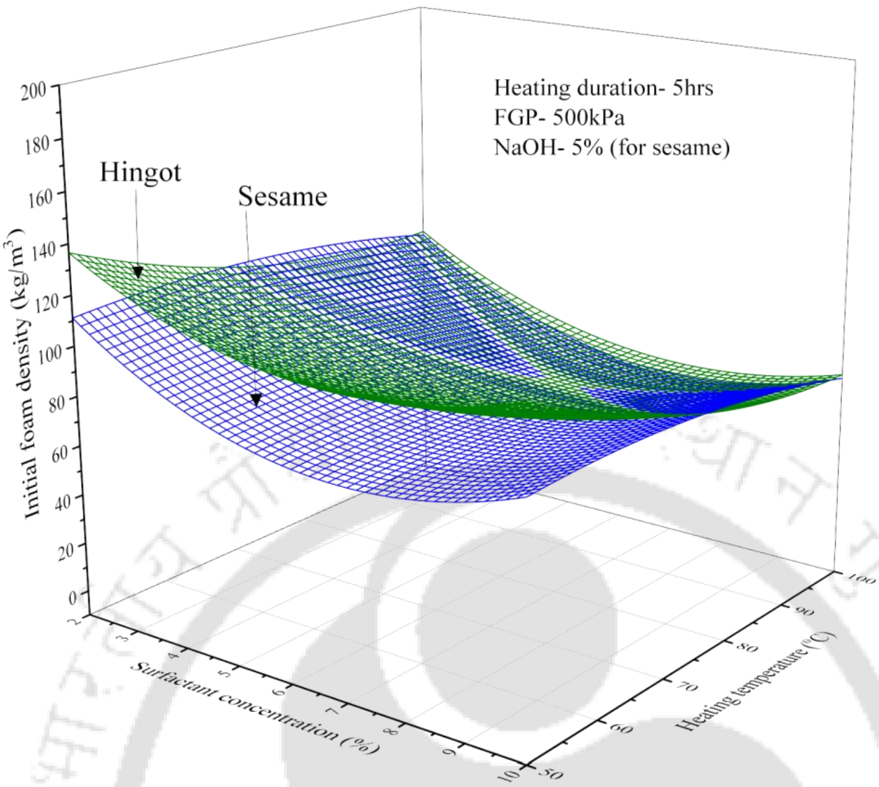


Figure 4-16 Variation of IFD with surfactant concentration and heating temperature

In line with this discussion, RSM plot on the effect of variation of NaOH concentration shows that, for a constant surfactant concentration, an increase in NaOH concentration from 2 to 6% results in a significant decrease of 38% in IFD due to enhanced hydrolysis of protein surfactant with NaOH (Figure 4-18). However, beyond the NaOH concentration of 6 %, due to a significant increase in demand for NH_4Cl solution for neutralization, it becomes detrimental as the quality of foam is affected. The negative impact on the quality of foam is evident from a substantial increase in IFD beyond NaOH concentration of 6%. Adding to the above, in line with earlier discussion on viscosity, the interaction effect between surfactant concentration and NaOH concentration on IFD is significant with $p < 0.05$ (Table 4-8).

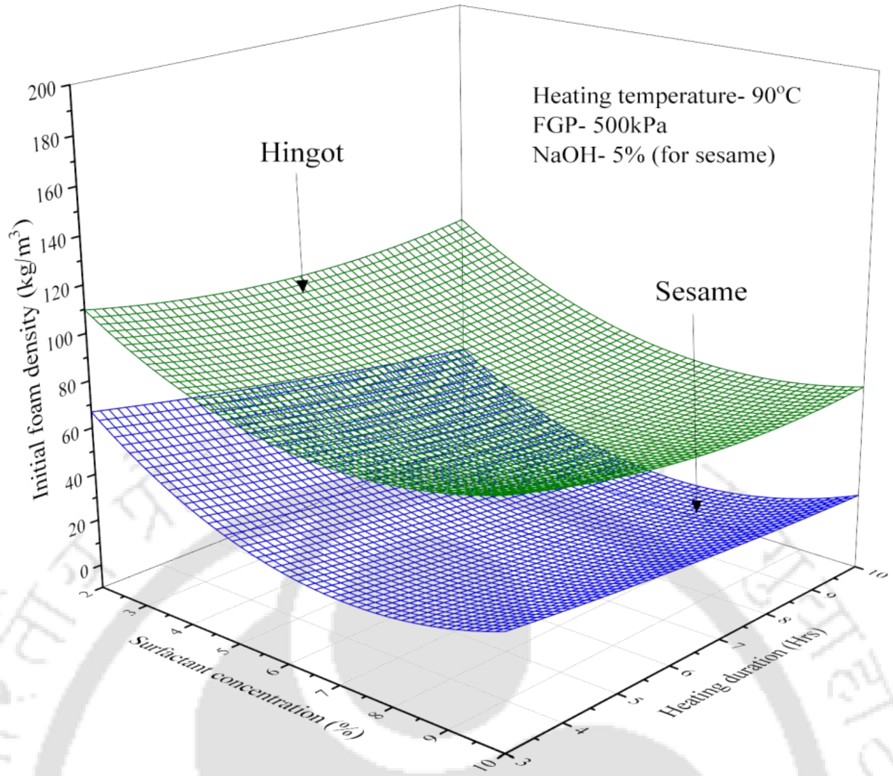


Figure 4-17 Variation of IFD with surfactant concentration and heating duration

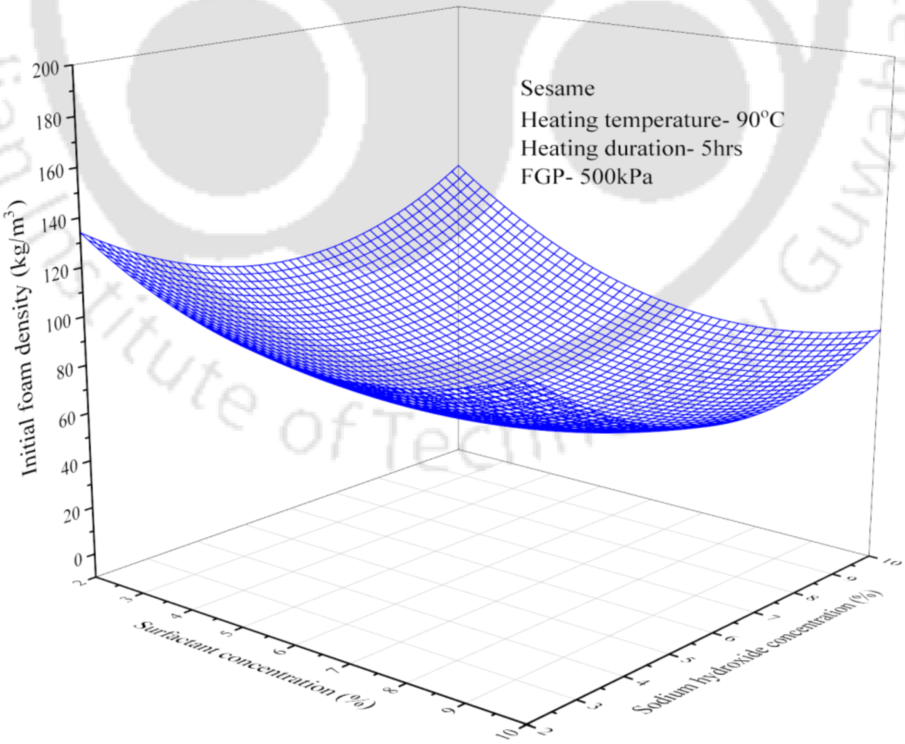
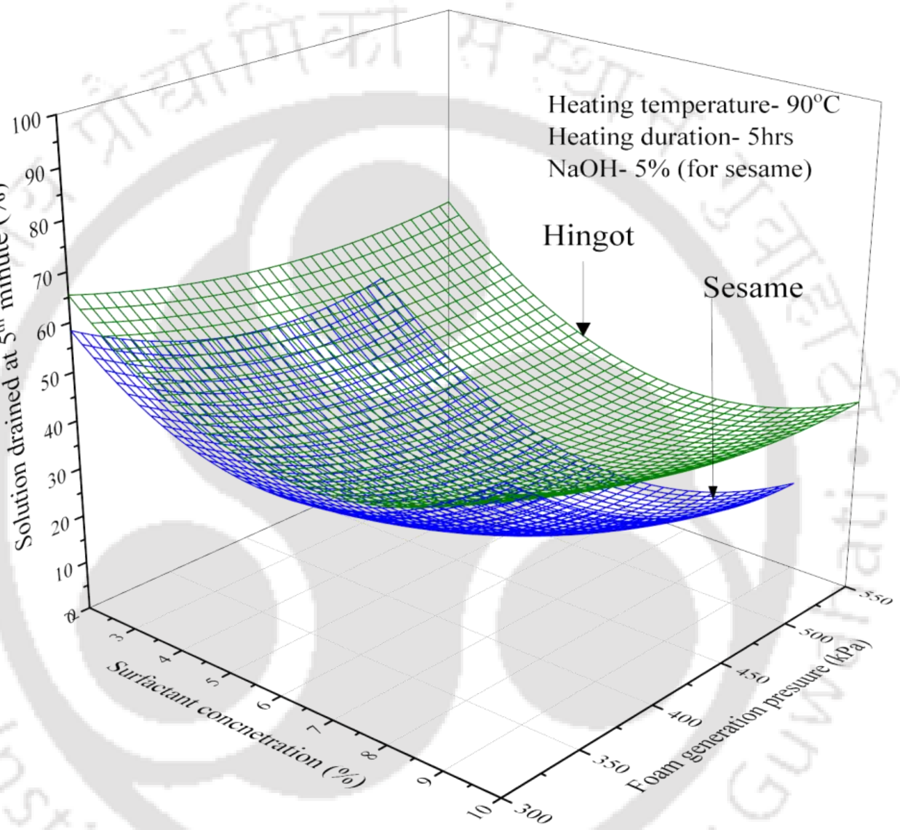


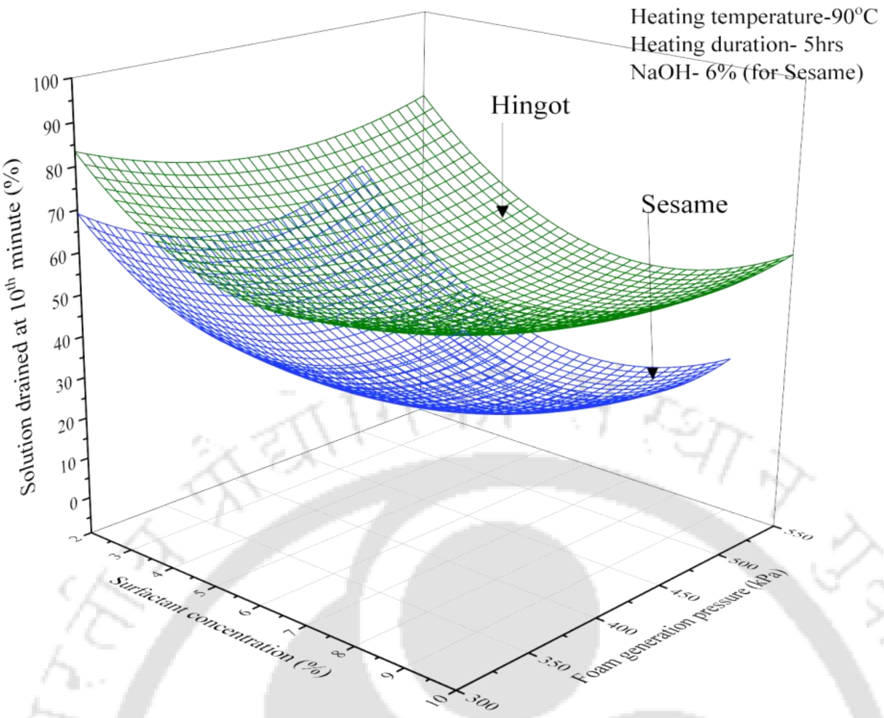
Figure 4-18 Variation of IFD with surfactant concentration and NaOH concentration

4.4.4 RSM model for foam stability

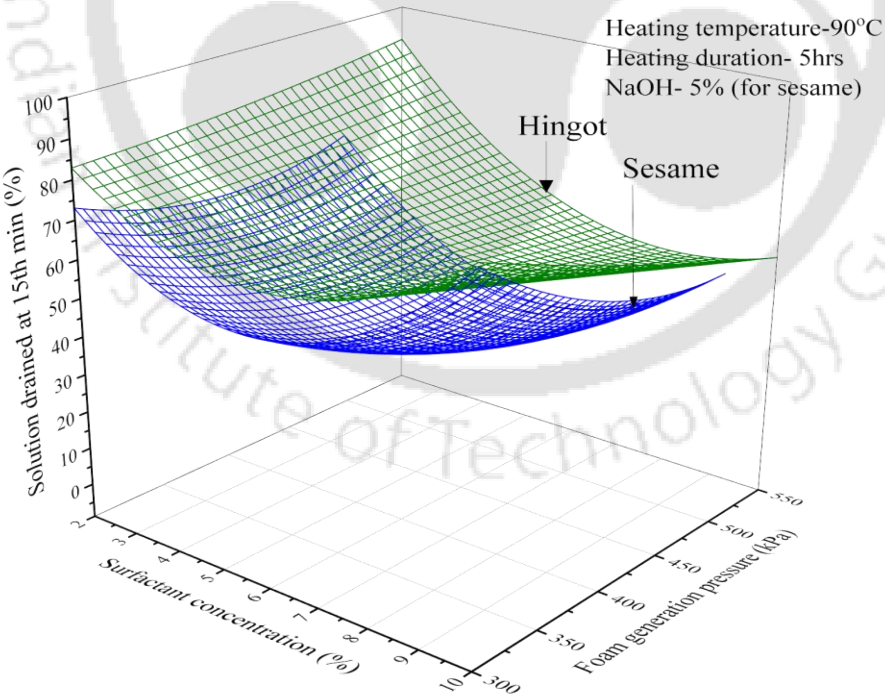
Foam stability is inferred from the variation of percentage solution drained with time. Results of ANOVA show that the main effects of all independent variables are significant (Table 4-7 and 4-9). Figure 4-19 depicts the RSM plots for percentage solution drained for hingot and sesame surfactant foams at several time intervals after foam generation. Foam drainage decreases dramatically with increasing FGP for both surfactants, similar to previous results on the influence of FGP on IFD. For instance, for sesame surfactant at higher surfactant concentration of 10%, a significant reduction of 49% in foam drainage (in 5 minutes) is experiential for increase in FGP from 300 to 500 kPa. The corresponding value for hingot surfactant is 29%. This result is an indication that adoption of higher FGP produces improved quality foam with lesser liquid fraction and less drainage. Evidently, for all concentrations investigated in this study, the foam drainage increases with time and the quantum of drainage is found to be relatively higher for less viscous hingot foam when compared to sesame foam. The plotted graphs show that, increase in surfactant concentration results in reduction in drainage till surfactant concentration of 7%. As the liquid fraction of foam reduces with increase in SC up to 7%, subsequently there is reduction in foam drainage also. Hence when SC is less than 7%, as the foam is wet, with increase in viscosity, reduction in foam liquid fraction and corresponding reduction in foam drainage is observed. .The clear foam drainage profile is elucidated in Figure 4-19 (a) which shows an initial sharp reduction in drainage till surfactant concentration of 5% followed by a plateau region with steadied value till 7%. However, beyond inflection point of 7%, in line with results on IFD, increase in drainage is observed due to increase in liquid fraction of foam. Further, the reduction in drainage with increase in surfactant concentration is more predominant at higher level of FGP which results in better quality of foam with reduced liquid fraction. The variations impact in heating temperature of sesame surfactant solution from 50 to 100^{o}C indicates a reduction of 25% in foam drainage for surfactant concentration of 10% (Figure 4-20 (a)). Further the effect of temperature is more evident on early foam drainage. On the other hand, the corresponding effect of temperature on hingot foam drainage is not substantial. On a similar note, increase in heating duration of surfactant solution from 3 to 10 hours resulted in a maximum reduction of 35% in foam drainage for sesame surfactant highlighting the impact of heating process on foam quality (Figure 4-21). In addition, there is found to be significant interaction between heating temperature and heating duration for sesame surfactant as represented graphically (Figure 4-22). Improvement of viscosity of surfactant solution with heating can be attributed to improvement of foam quality with reduced drainage.



(a) 5th minute

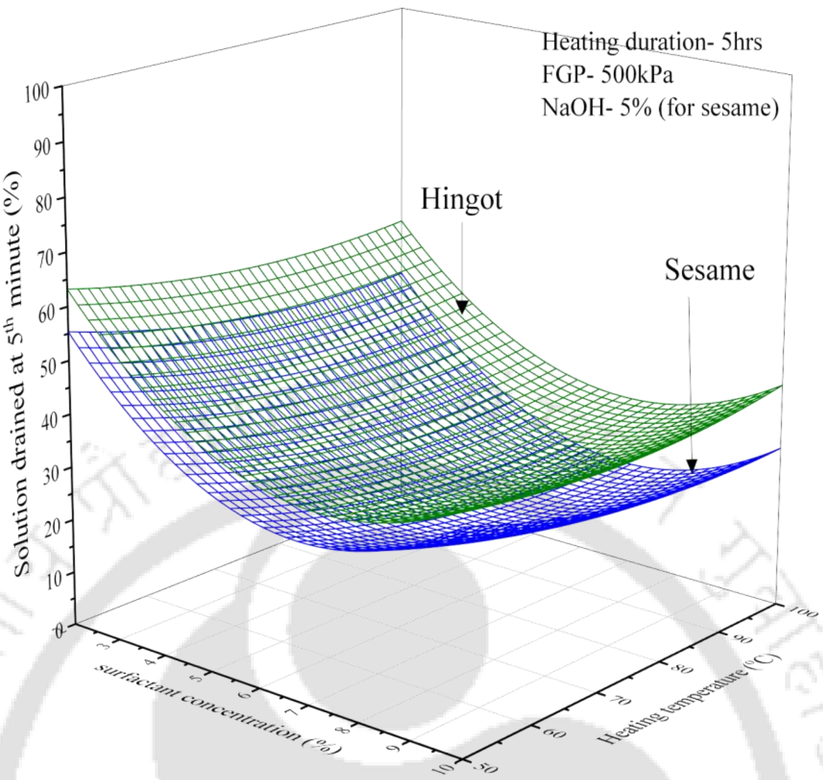


(b) 10th minute

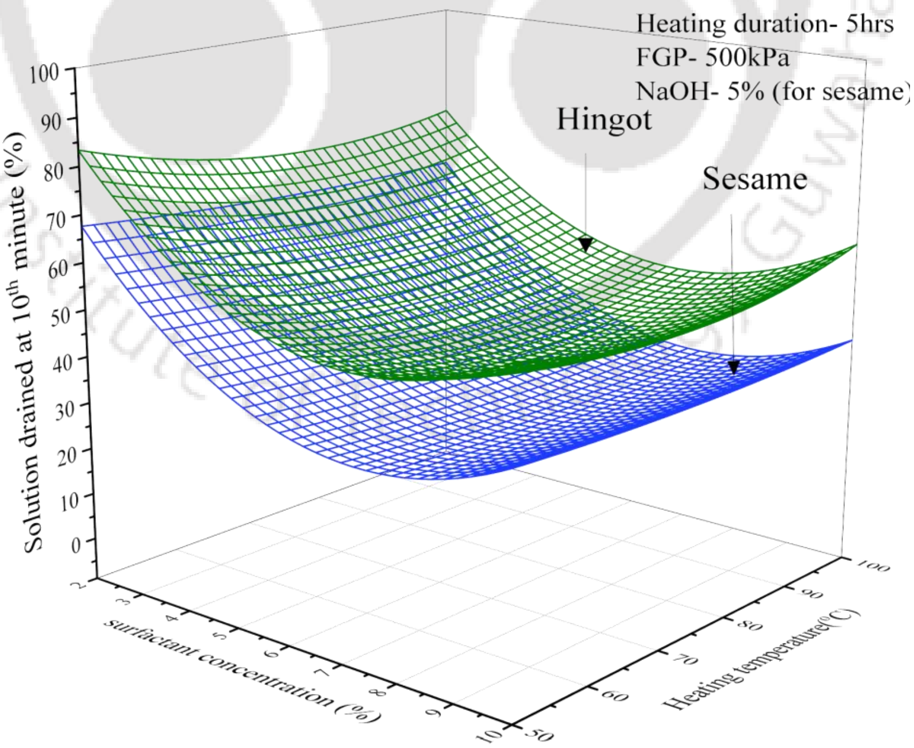


(c) 15th minute

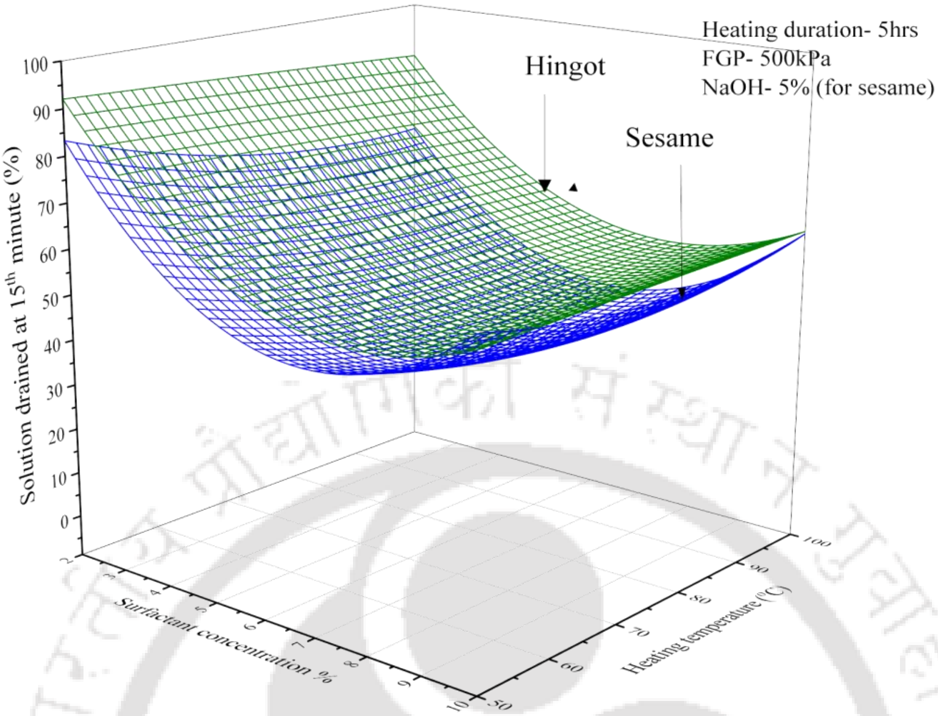
Figure 4-19 RSM plots for percentage solution drained for variation in surfactant concentration and FGP



(a) 5th minute

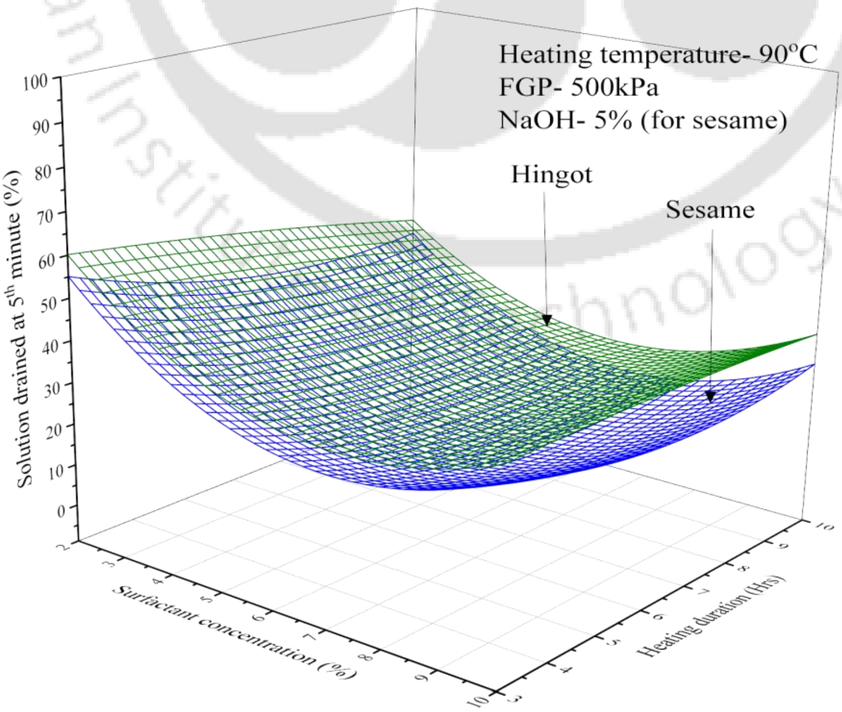


(b) 10th minute

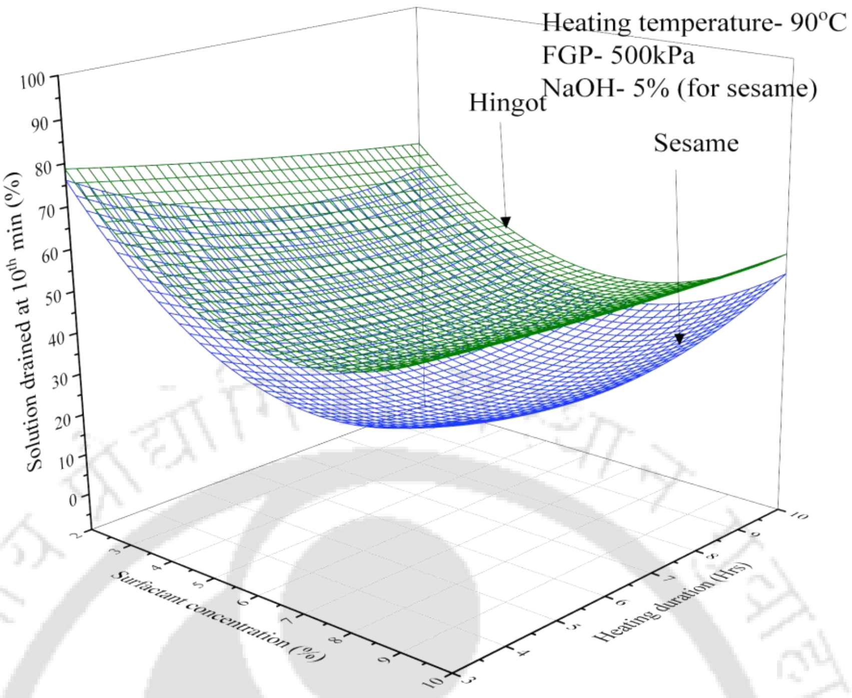


(c) 15th minute

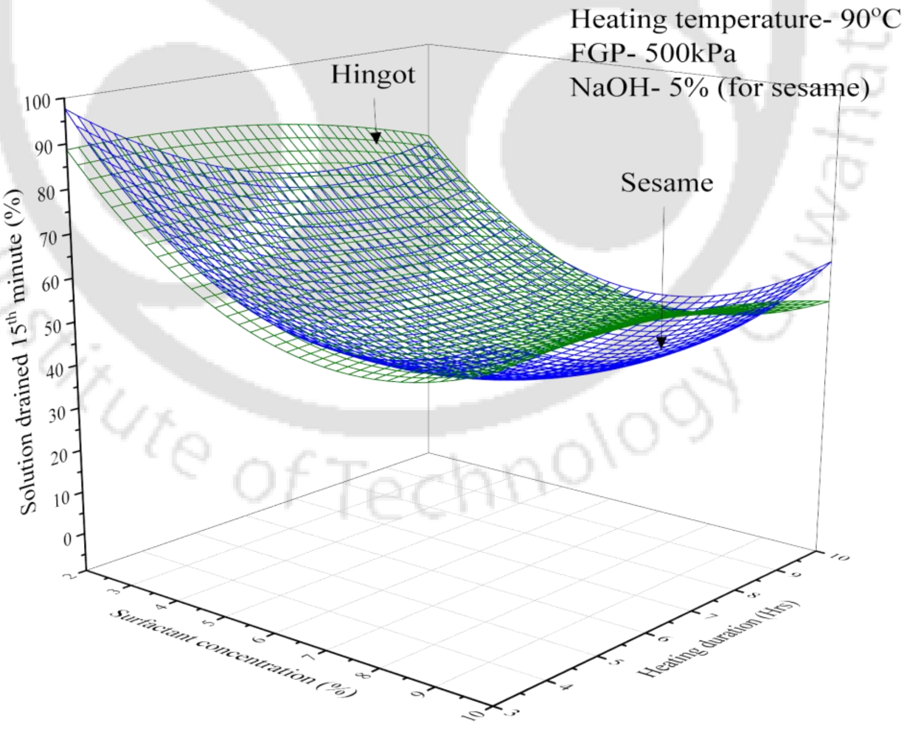
Figure 4-20 RSM plots for percentage solution drained for variation in surfactant concentration and heating temperature



(a) 5th minute



(b) 10th minute



(c) 15th minute

Figure 4-21 RSM plots for percentage solution drained for sesame surfactant with variation in heating duration

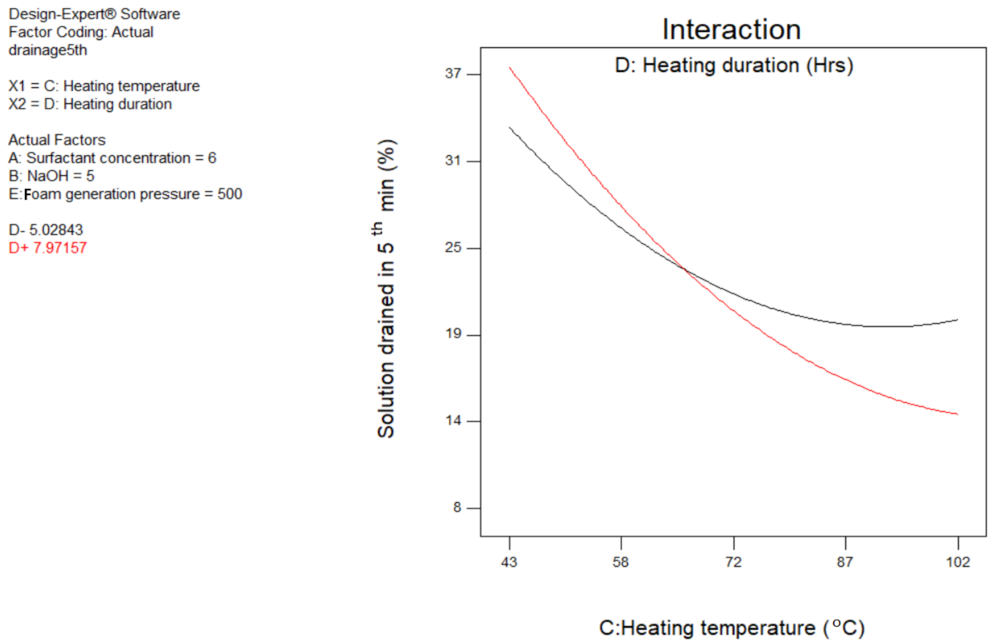


Figure 4-22 Heating temperature-heating duration interaction graph for response solution drained (sesame surfactant)

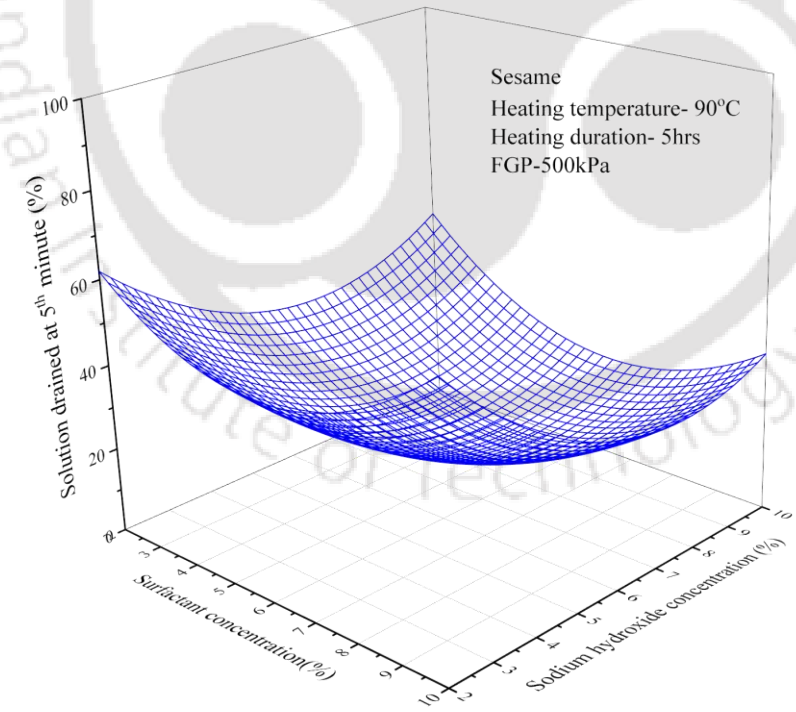


Figure 4-23 RSM plots for percentage solution drained for sesame surfactant with variation in NaOH concentration

Increase in NaOH concentration from 2 to 6% results in 26% and 49% decrease in drainage at lower surfactant concentration (2%) and higher surfactant concentration (10%) respectively (Figure 4-23). Enhancement in protein hydrolysis due to increase in NaOH concentration contributes to reduced foam drainage. However, beyond 6% NaOH concentration, increase in drainage is observed due to deterioration in foam quality resulting from greater demand of NH_4Cl solution for neutralization. Hence it can be inferred from the results that, within the range of parameters studied, the variation in foam drainage is found to be similar to IFD.

4.4.5 RSM model for foam bubble microstructure

ANOVA analysis shows that the main effects of all independent variables have significant effect on bubble size (Table 4-7 and 4-9). RSM plots for bubble size indicate that increase in FGP from 300 to 500 kPa results in considerable reduction in average bubble diameter (Figure 4-24). The above results are consistent with earlier literature (Sahu and Gandhi, 2021; Sheng, 2013). Further, results of statistical analysis prove the above notion as the F ratio of main effect FGP is very high particularly for sesame surfactant indicating significant influence of FGP on bubble size (Table 4-7). Adding to above, the trend of variation is found to be linear for hingot surfactant and nonlinear for sesame surfactant. Similarly, the correlation graph (Figure 4-25) indicates that increase in hingot surfactant concentration from 2 to 10% results in significant reduction of 47% in average bubble size which can be attributed to increase in viscosity of surfactant solution. This observation is very consistent with the literature, that surfactant solution with a high viscosity produces a stable foam with reduced drainage rate and smaller size foam bubble microstructure (Sahu and Gandhi, 2021; Sheng, 2013). However, for sesame surfactant, at higher FGP of 500 kPa, the effect of variation of surfactant concentration on bubble size is relatively less indicating significant interaction ($p<0.05$) between surfactant concentration and FGP (Table 4-8). Considering the effect of temperature, a maximum of 44% reduction in average bubble size is observed with increase in temperature from 50 to 100°C (Figure 4-26). These results suggest that enhancement in viscosity of surfactant solution with increasing temperature eventually reduces foam bubble size and bubble drainage. The above trend is evident in the correlation graph (Figure 4-25). Though sesame surfactant is highly viscous when compared to hingot, the experimental outcomes on bubble size do not show significant difference in their bubble sizes for most of the combination of parameters studied. This incident can be attributed to the fact that the foam images are captured immediately and hence the actual impact of viscosity on reduction of bubble size through prevention of bubble coarsening which happens at later stage is not reflected in the data. However, the above effect will be evident in the concrete microstructure analysis. On a similar note, the effect of increase in heating duration resulted in significant reduction in bubble size and this effect is more pronounced for sesame surfactant (Figure 4-27 and 4-28). The above observation is supported by earlier discussion on foam drainage at 5th minute wherein the effect of heating duration is predominant for sesame. Further, similar to observations on viscosity, there is not much effect on bubble microstructure due to variation of NaOH concentration (Figure 4-29).

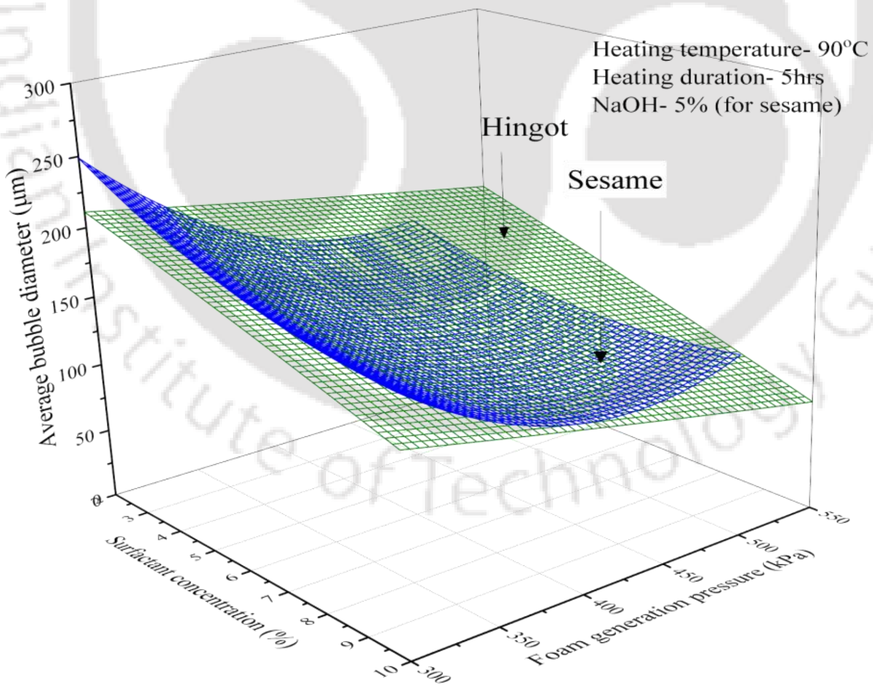


Figure 4-24 RSM plots for average bubble diameter for variation in surfactant concentration and FGP

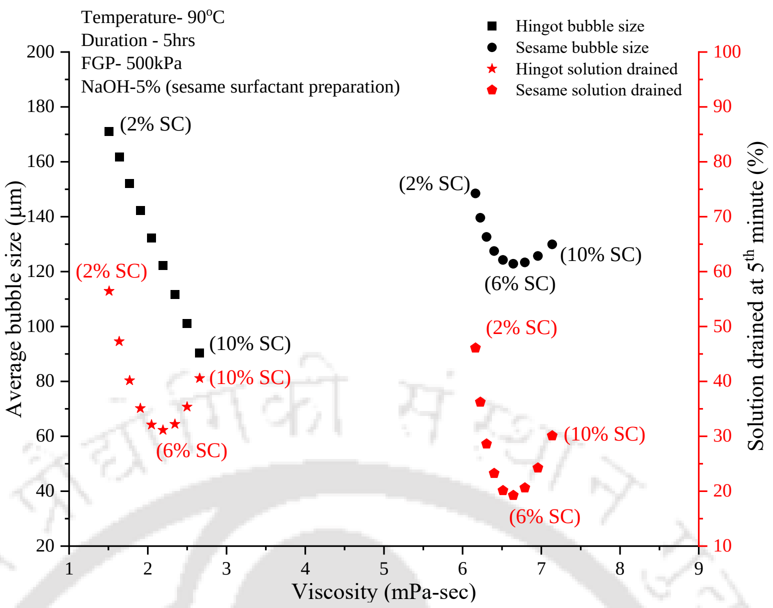


Figure 4-25 Relationship between viscosity, average bubble size and solution drained

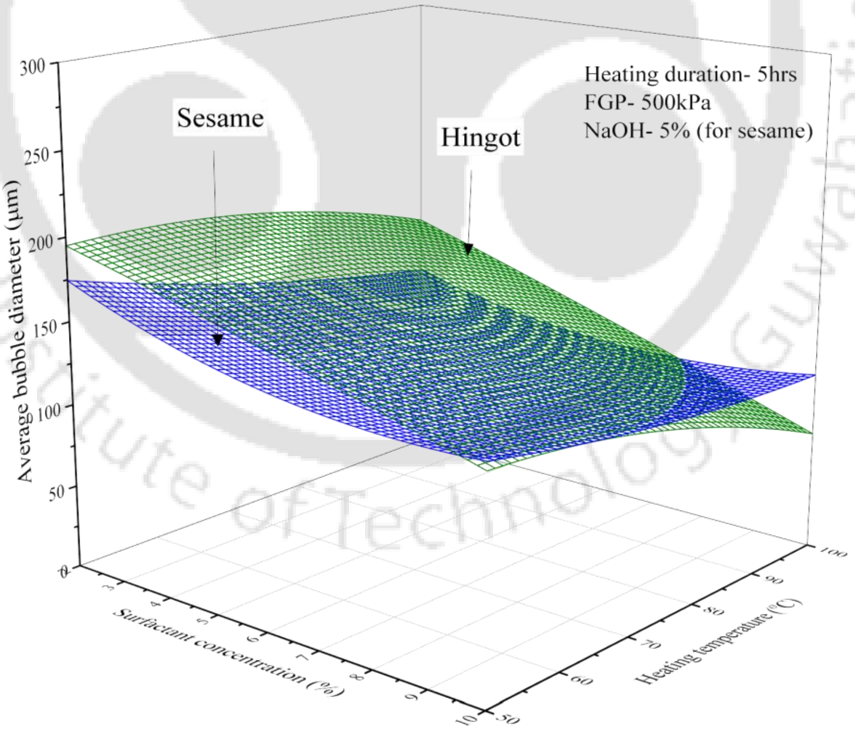


Figure 4-26 RSM plots for average bubble diameter for variation in surfactant concentration and heating temperature

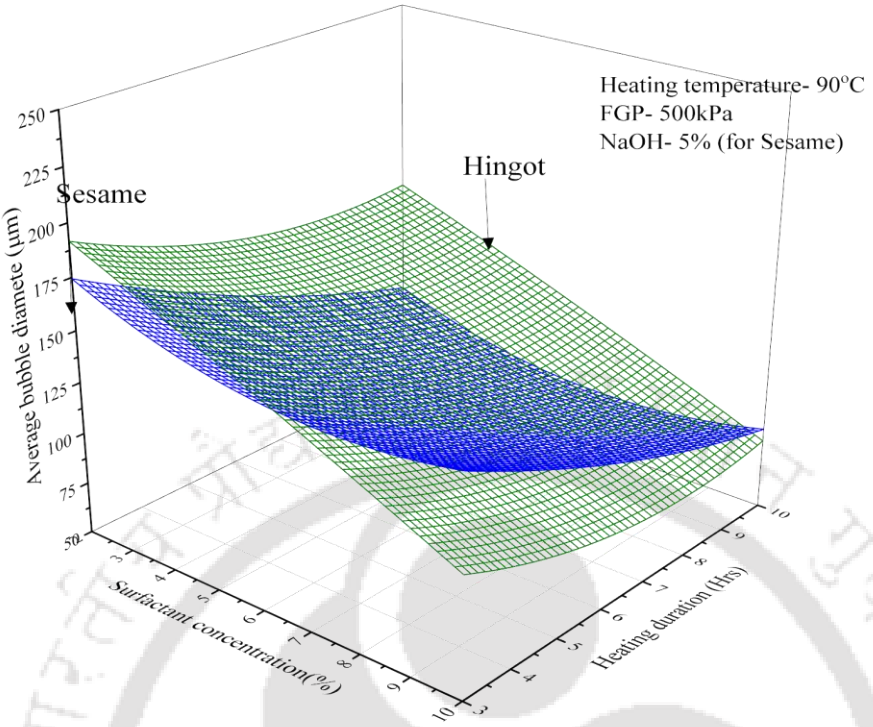


Figure 4-27 RSM plots for average bubble diameter for variation in surfactant concentration and heating duration

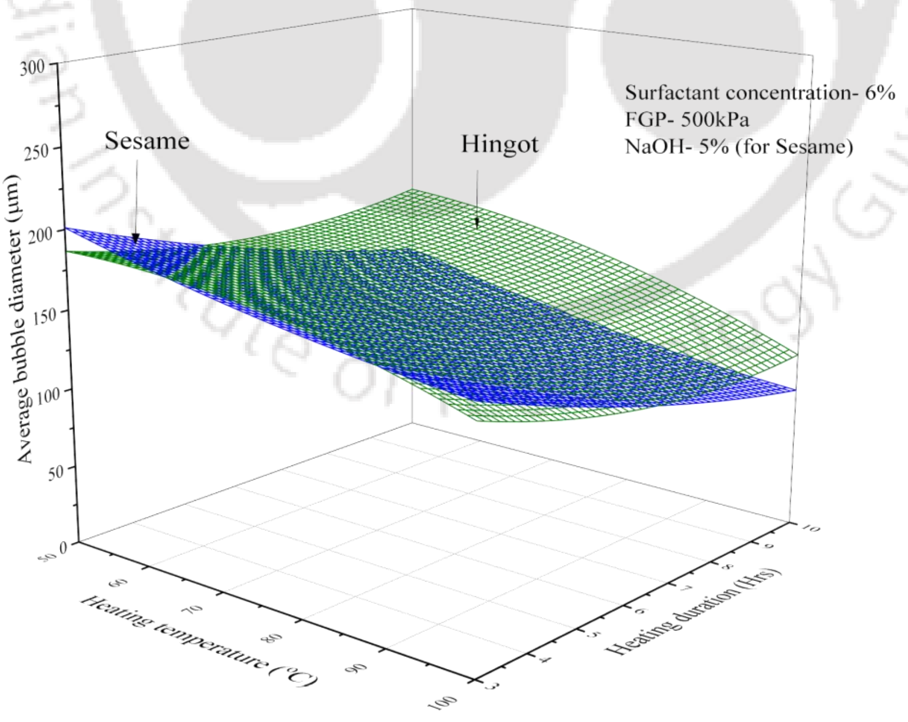


Figure 4-28 RSM plots for average bubble diameter for variation in heating temperature and heating duration

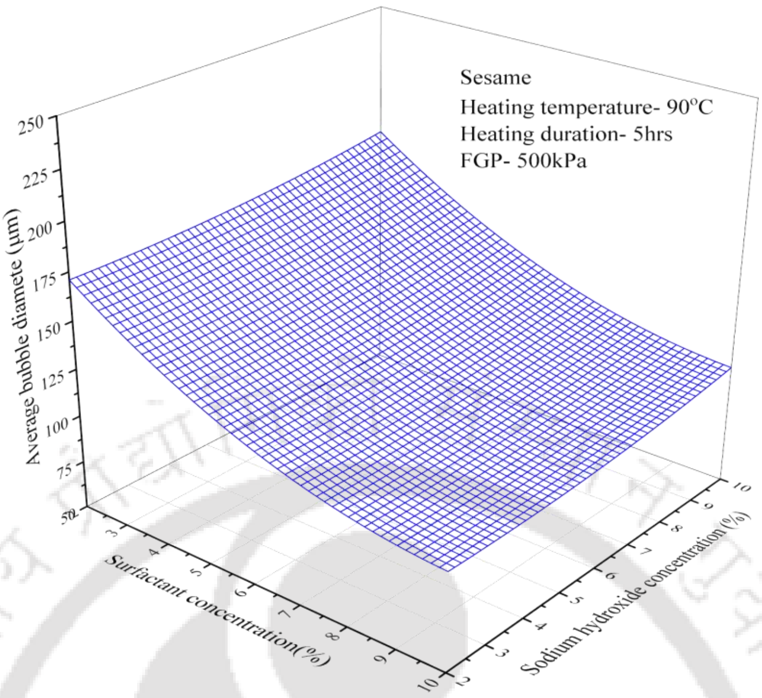
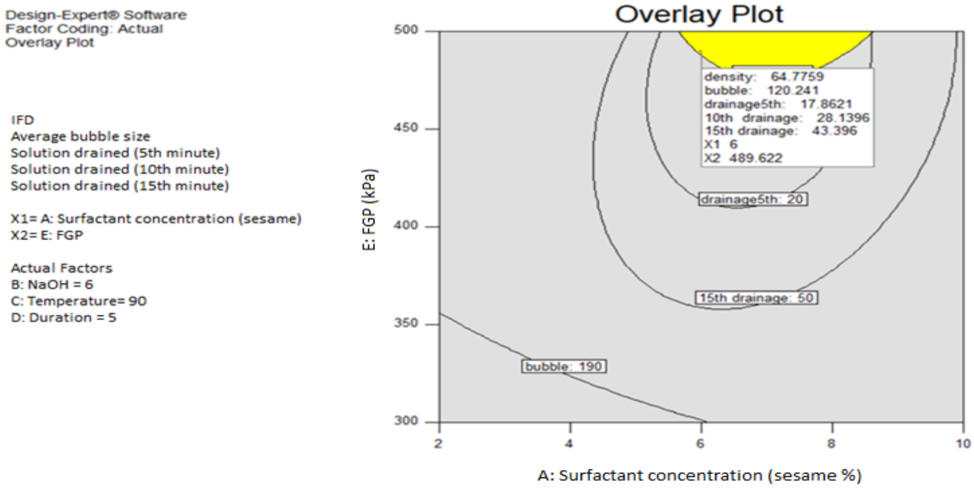


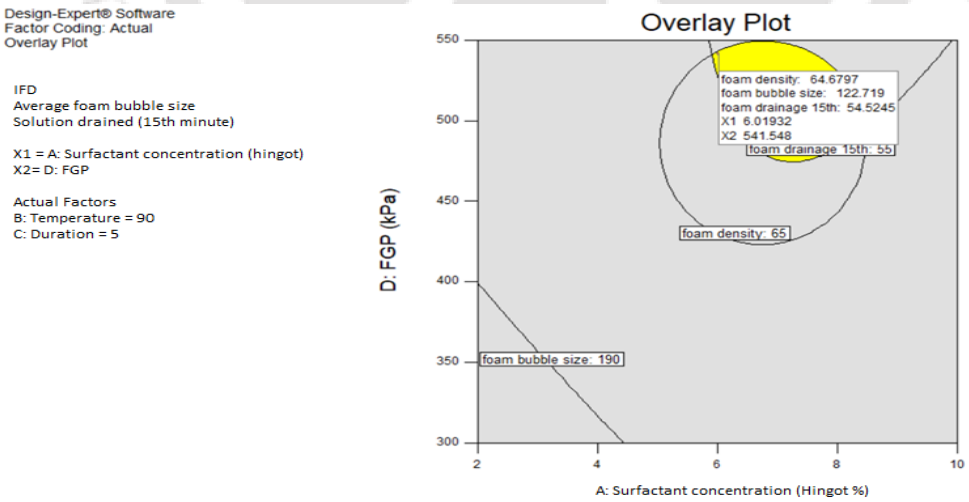
Figure 4-29 RSM plot for average bubble diameter for variation in surfactant concentration and NaOH concentration

4.5 Optimization of RSM

Optimization of various input parameters of surfactant extraction and foam production processes are needed to improvise the quality of foam. Multiple optimization approach is adopted using Design Expert with different constraints set for various parameters as mentioned below. As per ASTM requirement, the desired range of IFD is fixed as 30 to 65 kg/m^3. Similar to assumptions made in earlier studies (Sahu and Gandhi, 2021), the maximum permissible drainage in 15 minutes is fixed as 55 %. The range of desired surfactant concentration, NaOH concentration, heating temperature, heating duration and FGP are fixed as mentioned above in section on experiment design. RSM approach predicted the optimum combination of input parameters for sesame surfactant as 6% surfactant concentration, 490 kPa FGP, 6% NaOH concentration, 90°C heating temperature and 5hours heating duration. Similarly, for hingot surfactant the predicted parameters are 6% surfactant concentration, 550 kPa FGP, 90°C heating temperature and 5 hours heating duration. From the optimization study, it is evident that there is not much betterment in foam quality with increase in surfactant concentration beyond 6% and hence optimized surfactant concentration sounds be reliable. Further, at the optimized input parameters, the desirability value is found to be maximum and the responses of sesame and hingot surfactant are comparable as shown in Figure 4-30. Furthermore, it is to be noted that the above mentioned optimized parameters can differ if the raw material for surfactant extraction is procured in different seasons. Hence, in order to reduce the variations in the present study, the raw material for the entire study is collected in November season and procurement is made from one vendor.



(a) Sesame surfactant



(b) Hingot surfactant

Figure 4-30 Overlay plot for optimization process (Yellow colour indicates recommended zone of operation to get desired response)

4.6 Performance of Surfactants in Foam Cement Paste

Having optimised the surfactant and foam production parameters, as a next step, the performance of foam in cement paste needs to be checked. As per guidelines of ASTM C869, foam cement paste of design density 640 kg/m^3 is made with water to cement ratio of 0.58. Firstly, the homogenous base mix of cement-sand slurry is prepared in horizontal shaft paddle mixer. Then the calculated amount of foam produced at optimised parameters is added to the base mix and thoroughly mixed for 5 minutes to obtain homogenous mix. To study compressive strength, six number of 50 mm size cubes are cast, water cured for 25 days, followed by air drying for 3 days and then tested. Similarly, water absorption test is carried out on 50 mm cubes as per guidelines of ASTM C 642-21. As reported in earlier studies, considering the significant variation in density of foam concrete, water absorption is expressed as % by volume (Nambiar and Ramamurthy, 2007b). Table 4-13 shows that the foam cement paste produced with both the surfactants meets the ASTM requirements confirming the foam stability. Further, as the commercialization of any product is heavily dependent on its cost, rough estimates of the material costs are presented to assess its competitive position concerning similar products on the market. Analysis indicates that the costs of sesame seed and hingot fruit for one cubic metre of foam concrete (target density 1500 kg/m^3) are ₹400 and ₹60 respectively. In comparison to commonly available synthetic surfactants in the market, the proposed natural surfactants, particularly hingot, offer a much greater economic advantage.

Table 4-13 Properties of foam cement paste

Properties of foam cement paste	**Experimental results**		**Standard deviation**		**CoV**		**ASTM C869-11 requirements**
	Sesame	Hingot	Sesame	Hingot	Sesame	Hingot	
Fresh density (kg/m^3)	644	641	2.51	1.89	0.038	0.029	640 ± 50

Properties of foam cement paste	Experimental results		Standard deviation		CoV		ASTM C869-11 requirements
	Sesame	Hingot	Sesame	Hingot	Sesame	Hingot	
Dry density (kg/m^3)	461	445	6.53	4.82	0.014	0.011	490± 40
Compressive strength (MPa)	1.6	1.5	0.19	0.16	0.118	0.106	1.4 MPa
Water absorption (% by volume)	20.33	22.21	0.64	0.998	0.031	0.044	25 (max)

4.7 Summary

The surfactants extracted from two different natural sources (sesame and hingot) were investigated as prospective supplementary alternative to conventional synthetic foaming agents for use in foam concrete. A systematic study on variation of important foam and surfactant characteristics such as initial foam density, foam stability, foam bubble microstructure and viscosity of surfactant solution with surfactant and foam production parameters were carried out through experiment design based on response surface methodology. The possible response surface models have been developed for prediction of the foam and surfactant characteristics and their validity were verified experimentally. Optimization of various input parameters of surfactant extraction and foam production processes are carried out. Further the suitability of foam in production of concrete was assessed based on the performance of foam cement paste as per ASTM requirements.

Chapter 5

Effect of Type of Foaming Agent and Water to Solids Ratio on Foam Concrete Behaviour

5.1 General

The cellular structure in foam concrete which can be attributed to the various benefits of foam concrete is produced by adding suitable foaming agents which can be either synthetic based or natural based. Many studies have proved the fact that the performance of foam concrete produced with different foaming agents varies greatly (Liu et al., 2019; Falliano et al., 2018a; Sun et al., 2018; Tarameshloo et al., 2017; Tian et al., 2016; Panesar, 2013; Ranjani and Ramamurthy, 2010b). Further as the applications of foam concrete in building industry is increasing noticeably, more research interest is shown by researchers on enhancement of mechanical behavior of foam concrete. Although the mechanical behavior of porous material is expressed mainly as function of pore structure, some researchers have suggested that optimizing the solid structure can be considered as an alternative approach to production of foam concrete with better mechanical characteristics (Chung et al., 2020; Hilal et al., 2015b; Narayanan and Ramamurthy, 2000). In this regard, the effect of w/c ratio which is one of the critical parameters influencing the fresh state and hardened state properties of any type of concrete, need to be studied in a systematic manner for foam concrete also.

Based on the literature review it is evident that although the number of publications in the research area of foam concrete is steadily increasing, however the literature status indicates that as of now, the number of papers which are reported on the effect of water-solids ratio on the fresh state behaviour, microstructure and mechanical behaviour of foam concrete is very much limited. Having identified the major research gap, the main objective of present study is to carry out series of experimental investigations on the influence of water-solid ratio, on the foam concrete behaviour.

Firstly, investigations on the influence of water-solids ratio on the fresh state properties of foam concrete for mixes with different foam volumes and for two different natural surfactants namely sesame and hingot are studied. Then the influence of variation of water-solids ratio on the pore structure and the corresponding impact on the mechanical behaviour of concrete and water permeation characteristics are analyzed. The optimum water-solids ratio at which the fresh concrete characteristics, mechanical properties, and water permeation characteristics are satisfactory are derived for various densities of foam concrete. Further studies on microstructure are used to justify the experimental results. The optimized water-solids ratios derived for various mixes can serve as guidelines for future studies on optimization of performance of foam concrete. Further recommendation from this work could help to increase the utilization of recommended natural surfactants in the market gradually, which could present new sight in improving the environmental impact of foam concrete.

5.2 Materials and Mix Proportions

The materials used in these investigations are 43 Grade ordinary Portland cement (conforming to IS 269 with specific gravity of 3.15) and river sand (finer than 300μm with specific gravity 2.6). The particle size gradation and XRD pattern of river sand used for the present study are shown in Figure 5-1 and 5-2 respectively. The pre-foam method is adopted and foam is generated using foam generator based on compressed air method by aerating two different surfactant solutions extracted from sesame seed and hingot fruit. The details of extraction procedure of surfactant solution from sesame seed and hingot fruit is explained in details in previous chapter. The optimized surfactant extraction process and foam production parameters (as mentioned in section 4.5) viz., sesame surfactant concentration, foam generation pressure (FGP), NaOH concentration (alkali for hydrolysis), temperature of heating and duration of heating arrived at as 6%, 490 kPa, 6 %, 90^{o}C and 5 hours have been adopted in this section. Similarly, for hingot surfactant, the optimized surfactant extraction and foam production parameters of 6% hingot surfactant concentration, 550 kPa FGP, 90^{o}C of heating temperature and 5 hours of heating duration have been adopted. Table 5-1 presents the basic properties of foam and surfactant solution used in this study. All mixtures are designed with a fixed cement-sand ratio of 1:2 by weight. As of now, there is no standard method available for mix design of foam concrete. 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 cement sand ratio of 1:1 (Ranjani, 2011) and 1:2 (Nambiar,2006) for their studies. From economic point of view, a constant cement sand ratio of 1:2 has been adopted for the present study.

A series of foam concrete mixes with different target densities, namely 1000, 1500, and 1800 kg/m^3, are prepared by varying the foam volume between 52.8 % and 12 %. The details of mix formulations used in this experimental programme are summarized in Table 5-2. As the mixing procedure significantly affects the material property, the following procedure is followed consistently for all the mixes prepared. horizontal shaft paddle type mixer machine with speed of 40 rpm as recommended by ASTM C796 has been adopted in the present study. Also, a proper mixing sequence has been adopted to minimize foam damage. The mixing sequence consists of first preparation of homogeneous base mix of cement and sand slurry by mixing it for almost 4 min, followed by addition of calculated weight of foam to the base mix and further mixing it for 2 to 3 min in order to get uniform distribution of foam [Sahu et al., 2018]. In addition, to ensure that the foam damage is minimal, check on the initial fresh density of concrete is made. The measured plastic density of concrete is to be within ±50 kg/m3 of its target density as prescribed by ASTM C869.

Table 5-1 Properties of foam and surfactant

Raw material	**Appearance of surfactant solution**	**Surfactant Concentration (%)**	**FGP (kPa)**	**Initial foam density (kg/m^3)**	**Viscosity of surfactant solution (mPa.s)**	**pH value of surfactant solution**
Sesame seed	Brown	6	490	54	6.5	8 ±0.5
Hingot fruit	Dark brown	6	550	63	2.06	4.5 ±0.5

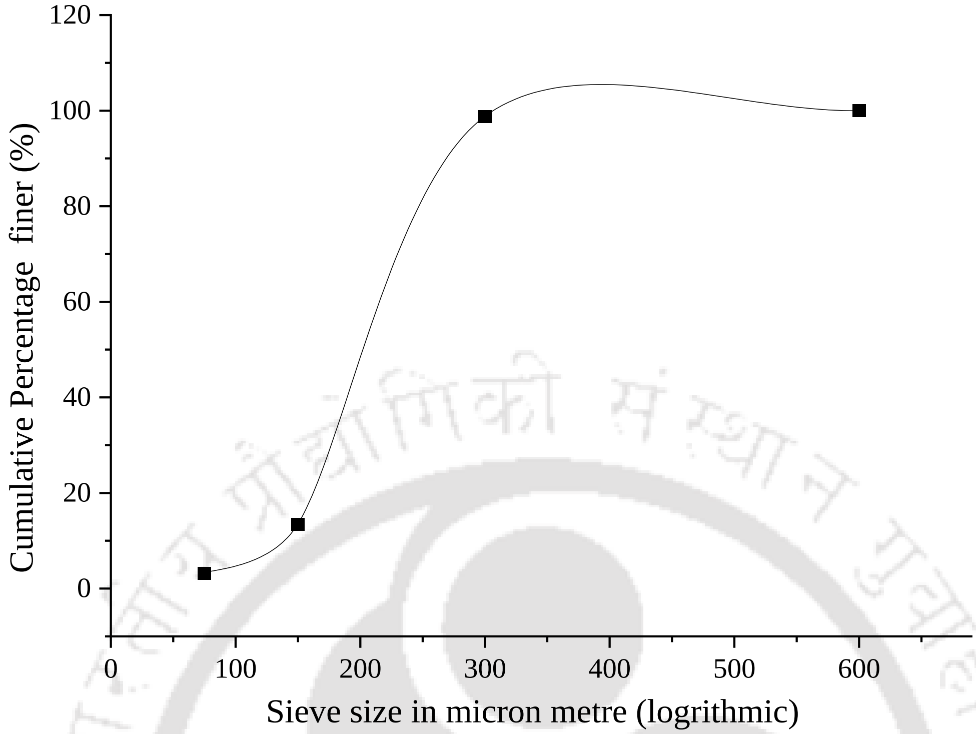


Figure 5-1 Grain size distribution of sand

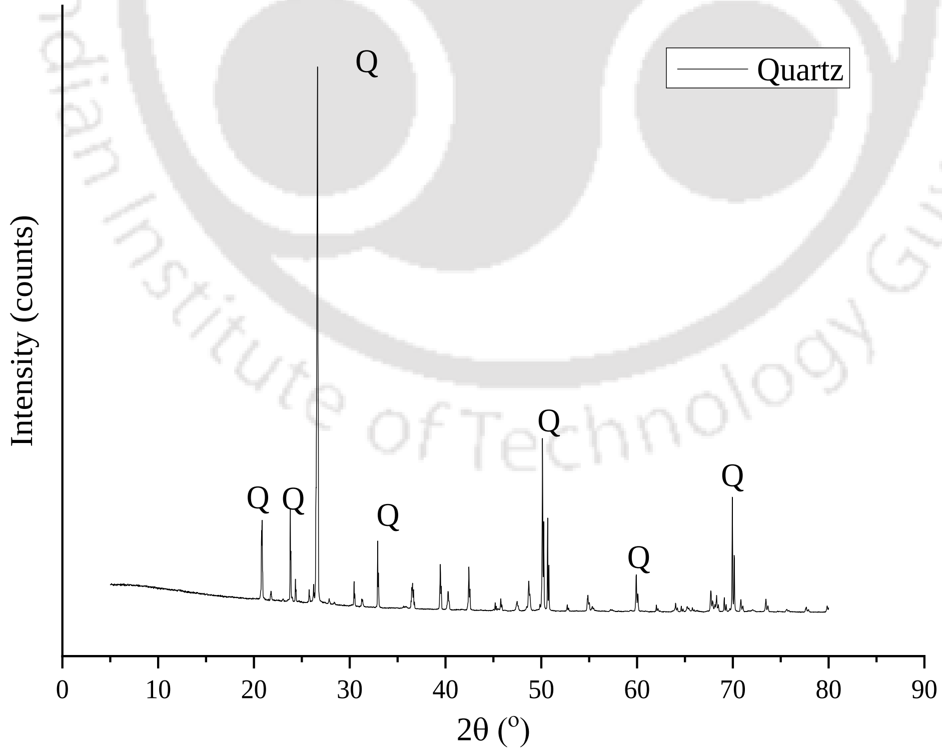


Figure 5-2 XRD pattern of sand

Table 5-2 Details of mix proportions of prepared cement- sand foam concrete mixes

Mix No.	Target fresh density (kg/m^3)	Cement (kg/m^3)	Fine aggregate (kg/m^3)	Water-solids ratio	Foam (kg/m^3)
SM 1	1000	271	542	0.23	29.82
SM 2	1000	266	533	0.25	29.35
SM 3	1000	260	520	0.28	28.65
SM 4	1000	256	512	0.3	28.21
SM 5	1000	252	505	0.32	27.79
SM 6	1000	246	493	0.35	27.17
SM 7	1000	241	483	0.38	26.58
SM 8	1500	406	813	0.23	16.19
SM 9	1500	400	800	0.25	15.47
SM 10	1500	390	781	0.28	14.45
SM 11	1500	384	769	0.3	13.78
SM 12	1500	378	757	0.32	13.14
SM 13	1500	370	740	0.35	12.21
SM 14	1500	362	724	0.38	11.33
SM 15	1800	487	975	0.23	8.01
SM 16	1800	480	960	0.25	7.15
SM 17	1800	468	937	0.28	5.91
SM 18	1800	461	923	0.3	5.12
SM 19	1800	454	909	0.32	4.35
SM 20	1800	444	888	0.35	3.24
SM 21	1800	434	869	0.38	2.18
HM 1	1000	271	542	0.23	35.13
HM2	1000	266	533	0.25	34.56

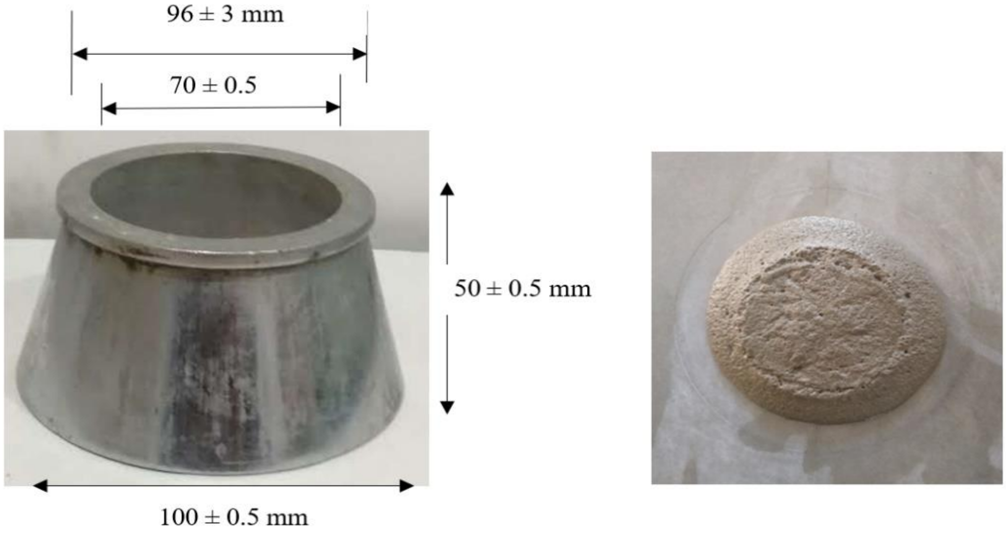
Mix No.	Target fresh density (kg/m³)	Cement (kg/m³)	Fine aggregate (kg/m³)	Water-solids ratio	Foam (kg/m³)
HM 3	1000	260	520	0.28	33.75
HM 4	1000	256	512	0.3	33.23
HM 5	1000	252	505	0.32	32.73
HM 6	1000	246	493	0.35	32
HM 7	1000	241	483	0.38	31.3
HM 8	1500	406	813	0.23	19.07
HM 9	1500	400	800	0.25	18.23
HM 10	1500	390	781	0.28	17.01
HM 11	1500	384	769	0.3	16.24
HM 12	1500	378	757	0.32	15.48
HM 13	1500	370	740	0.35	14.39
HM 14	1500	362	724	0.38	13.35
HM 15	1800	487	975	0.23	9.44
HM 16	1800	480	960	0.25	8.43
HM 17	1800	468	937	0.28	6.97
HM 18	1800	461	923	0.3	6.04
HM 19	1800	454	909	0.32	5.13
HM 20	1800	444	888	0.35	3.82
HM 21	1800	434	869	0.38	2.57
BM 1	1800	487	975	0.3	-
BM 2	1921	444	888	0.3	-
BM 3	1980	384	769	0.28	-

SM- Sesame foam concrete mix, HM- Hingot foam concrete mix, BM- Base mix

5.3 Test Methods

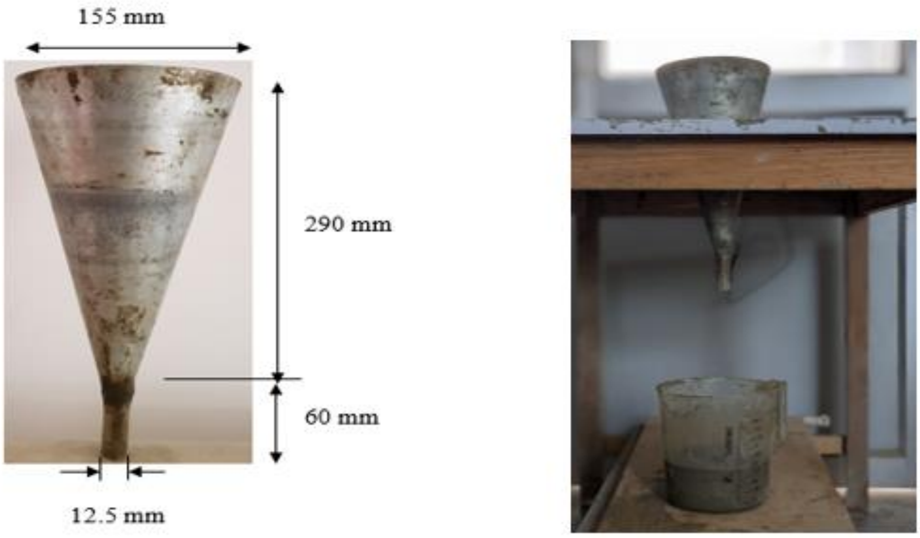
5.3.1 Fresh State Characteristics of Foam Concrete

Generally, fresh density of concrete is of utmost importance to determine the stability of foam in foam concrete (Nambiar and Ramamurthy, 2008a). To achieve stable mix, the measured plastic density of concrete is to be within ±50 kg/m^3 of its target density as prescribed by ASTM C869. Further for the present study, both the fresh and demoulded density of the casted specimens are considered to assess the stability of the foam concrete mix. The water-solids ratio needed to derive stable and workable foam concrete mixes with actual fresh and demoulded densities within ±50 kg/m^3 of the specified target density are determined. Following that, the workability of the foam concrete mixtures must be evaluated. The spreadability is measured using standard flow cone in accordance with ASTM C230 (Figure 5-3). After filling the cone with the mixture, it is elevated and the average flow of concrete is measured in four directions without shaking the flow table, which could impact the entrained air bubbles. The Marsh cone test is used to assess flowability, which is dependent on the fluidity of the mix (Figure 5-4). Firstly, 1.5 litres of sample is placed in the modified Marsh cone, and then it is timed to find how long it takes 1.0 litre of foam concrete to flow through the constricted orifice.



(a) Flow cone test mould (b) Spread

Figure 5-3 Flow cone test



(a) Modified Marsh cone apparatus b) Flow test

Figure 5-4 Modified Marsh cone test

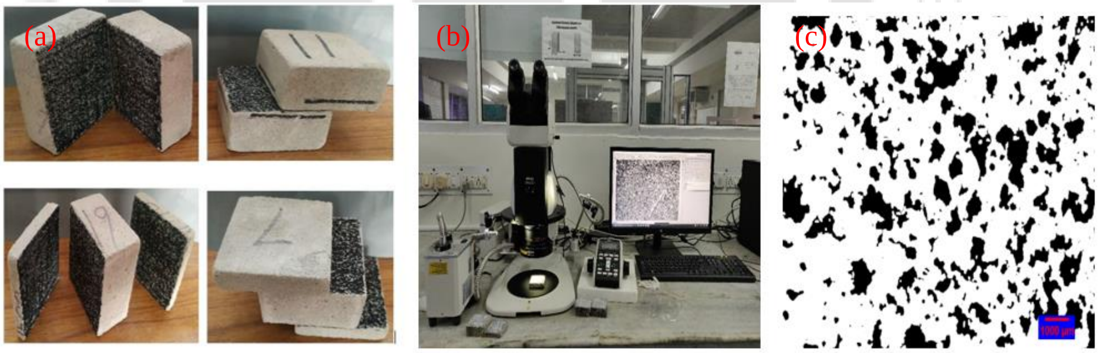
5.3.2 Air Void Characterization

The current study uses optical microscopy coupled with the image analysis method to study the air void characteristics of foam concrete. For the present study, four 50 mm cube samples are cast and moist-curing is adopted for 28 days for each density of concrete. Slices of dimensions 50 × 50 × 25 mm are extracted from cubes with a blade, parallel and perpendicular to the cast surface, from various depths of the specimen. For each combination, this slicing produces six specimens (cut surfaces).

Earlier studies have highlighted that traditional approach of filling pores with synthetic resins followed by smoothing with grinding is not compatible for foam concrete as the preparation process affect the physical structure of concrete (Petrov and Schlegel, 1994). As a result, the current investigation uses the ASTM C 457 specimen surface preparation procedure for contrast enhancement. The specimen surface preparation involves various steps viz., lapping of specimens with abrasive paper of fineness grade 1000, cleaning with compressed air, drying in oven at 50^{o}C for 3 days, applications of coats of black ink followed by filling of pores with white powder as discussed in detail in work by Sahu and Gandhi (2021). The prepared specimen with voids and remaining regions in contrasting colours is analyzed using optical microscope. The macropores of foam concrete with diameter more than 50µm are of prime interest in this study. A magnification of 0.7 X is chosen, with a pixel size of 0.162 microns established via calibration. For each foam concrete mix, a total of 36 images are taken at arbitrary positions on the six prepared surfaces (Figure 5-5). The Feret diameter, which is the largest distance between two parallel tangential lines drawn to the air void, is used to estimate the air void size distribution in concrete (Image J, 2006). For each mix, the circularity factors (F_{circ}) were calculated as a function of the perimeter and surface area of each pore ($F_{circ} = 4\pi$ (Area/Perimeter^2)). For a perfect pore, circularity equals 1; for irregular shapes, it is smaller.

5.3.3 Mechanical and Water Permeation Characteristics

To investigate the compressive strength and split tensile strength, six 50 mm cubes and three nos. of 100 × 200 mm cylinders are cast respectively (for each mix) and moist cured for 25 days and air cured for 3 days. The compressive strength and splitting tensile strength tests are performed according to ASTM C 796. For the water absorption test, 6 cubes are made for each mix. After being cured for 28 days, the specimens are oven-dried at 100°C to acquire the dry weight and then weighed after immersing in water for 24 hours after attainment of constant mass.



(a) Images of prepared specimens (b) Optical microscope (c) Binary image of hingot foam concrete- Design fresh density 1000 kg/m^3

Figure 5-5 Experimental set up for air void characterization

5.3.4 Mineralogical Analysis

An X-ray diffractometer is used for the present study where Cu Kα radiation of wavelength 1.54 Å is used for scanning at a voltage of 30 kV, current 30 mA and scanning speed of 1° per minute. Specimen of foam concrete from each mix (after 28 days of curing) is ground into powder form and thereafter passed through a 75 μm sieve. The intensity of the reflected X-rays is measured when the sample and detector are rotated throughout a 2θ angular range of 5° to 75°. The peak intensities at various angles are compared to the ICDD (International Centre for Diffraction Data) database, and the mineral phases of the samples are assessed using X-pert High Score Plus software (Version 3.0). Further, the elemental content of foam concrete mixtures is determined using the Energy Dispersive Spectroscopy technique.

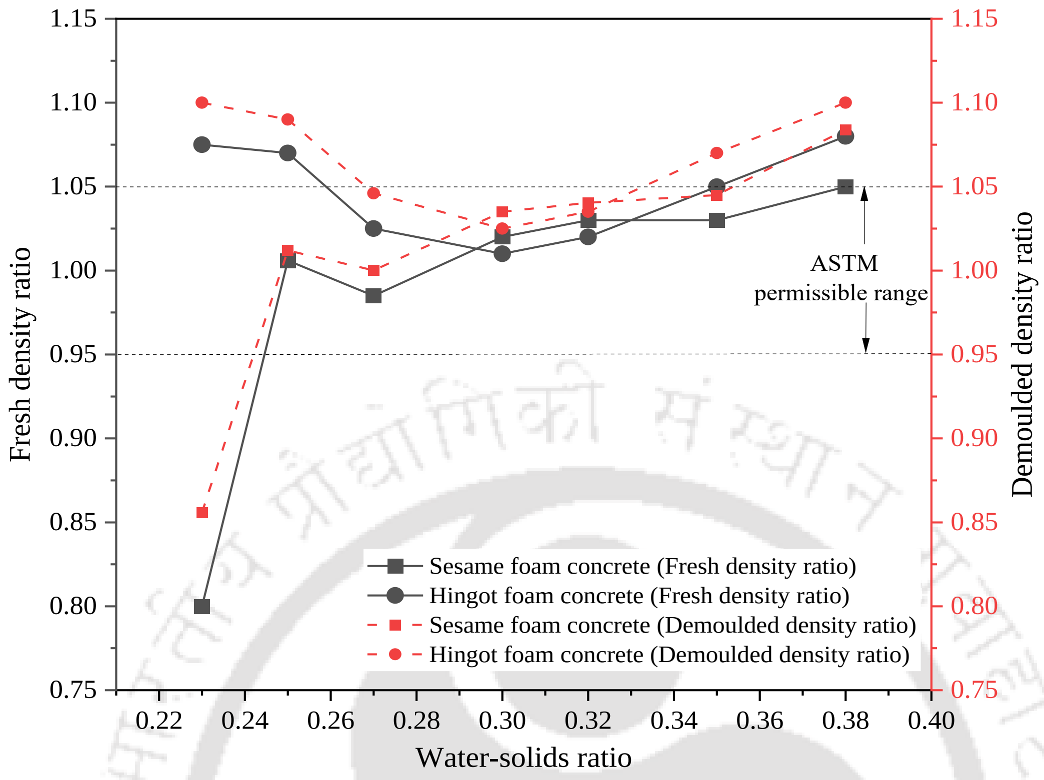
5.4 Results and Discussion

5.4.1 Effect of water-solids ratio on stability of foam concrete mixes

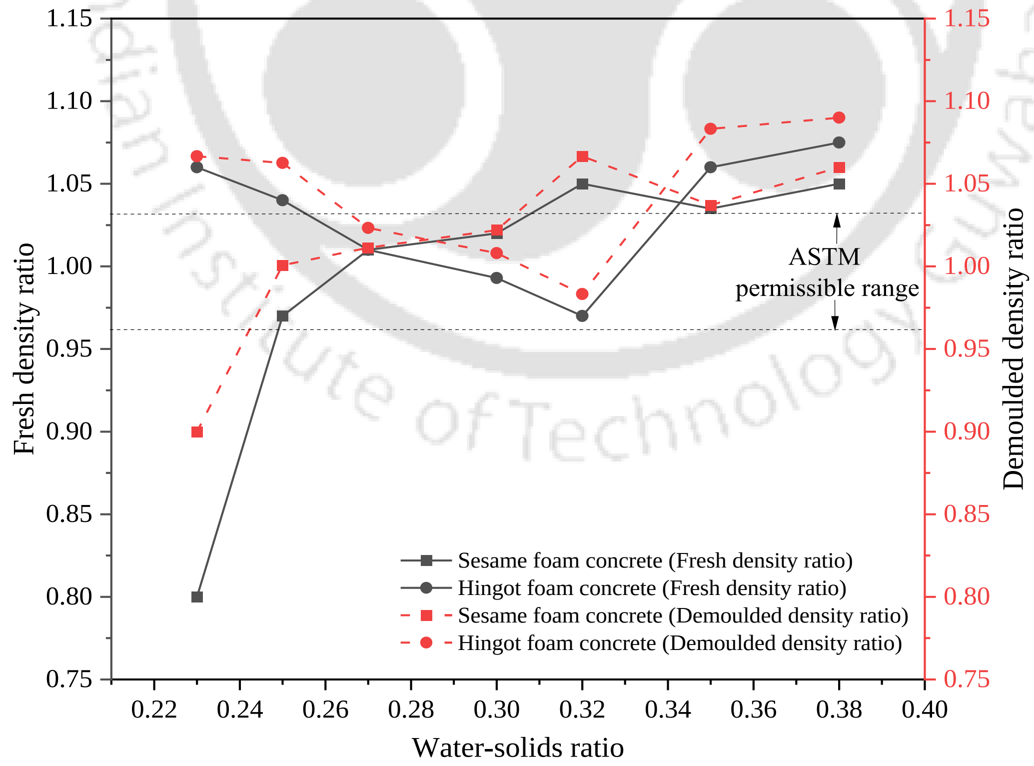
The experimental outcomes as represented graphically in Figure 5-6, indicate that for the sesame surfactant, the observed fresh density ratio and demoulded density ratio are very much less at lower water solids ratio of 0.23 when compared to that of hingot surfactant. The above observation is evident particularly for mixes with relatively higher foam volume (target density 1000 and 1500 kg/m^3) (Figure 5-6 (a) and (b)). Sesame surfactant being more viscous than hingot, at low water-solids ratio of 0.23, uniform blending of foam into the mortar cannot occur which results in density ratio to be significantly lower. However, when water-solids ratio is increased to 0.25, stable mixes are obtained. On the other hand, adopting a very high-water solids ratio of 0.38 also results in unstable mix with high density ratio and this can be attributed to the segregation of foam from mixes and collapse of foam at high water-solids ratio. The foregoing observation is consistent with previous studies findings that mixes are unstable at high water content (Sahu and Gandhi, 2021; Chen et al., 2020; Piotrowski and Prochoń, 2018; Chen et al., 2014b; Narayanan and Ramamurthy, 2012, 2008a). Further, it can be observed from the Figure 5-6 (c) that at some points though the fresh density requirement is met, the demoulded density is higher than ±50 kg/m^3 of the target density. Therefore, it is preferable to consider both fresh and demoulded densities for the stability assessment to determine the optimum range of water-solids ratio.

Comparative research on two types of surfactants shows that the fresh state characteristics of hingot foam concrete is found to be different from that of sesame, which can be attributed to the difference in nature of surfactant. At a very low water-solids ratio of 0.23, for hingot surfactant, the observed density ratio is very high, unlike sesame surfactant. This can be ascribed to the relatively stiff mixes which results in collapse of foam and increase in actual density of foam concrete. The above observation is supported by earlier studies which proved that addition of less water may result in rapid degeneration of foam due to lack of water for initial hydration (Jose et al., 2021; Sahu and Gandhi, 2021; Nambiar and Ramamurthy, 2012, 2008a). However, with increase in water-solids ratio, the density ratio reduces and falls within the range needed for stable mixes.

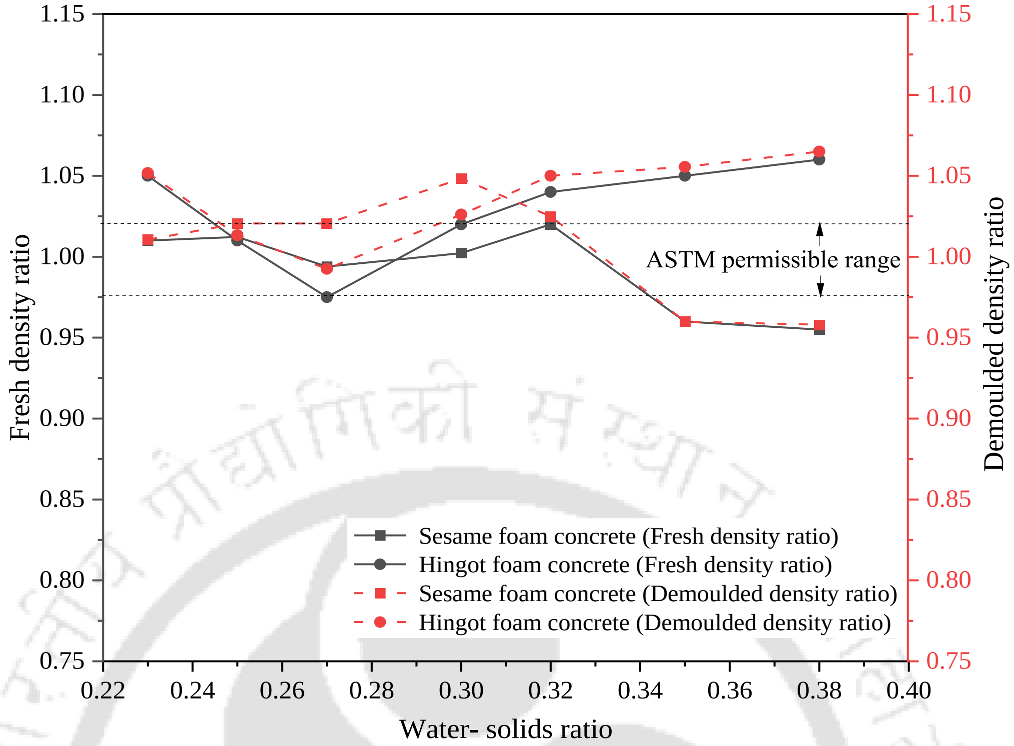
The optimum range of water-solids ratio for production of stable foam concrete mixes determined by trial and error for different target densities for two types of surfactants is presented in Table 5-3. From Table 5-3, it is evident that the water-solids ratio requirement for attaining stable mixes increases slightly with increase in foam volume. This is consistent with the findings of previous studies conducted by other researchers (Sahu and Gandhi, 2021; Nambiar and Ramamurthy, 2008a). Further, it is to be noted that the water- solids ratio requirement for stable foam concrete mixes is comparable for hingot and sesame surfactant. The values attained are in close agreement with water-solids ratio reported in earlier studies for natural surfactants (Nambiar and Ramamurthy, 2008a).



(a) Target fresh density 1000 kg/m^3



(b) Target fresh density 1500 kg/m^3



(c) Target fresh density 1800 kg/m^3

Figure 5-6 Variation of density ratio of foam concrete with the water-solids ratio

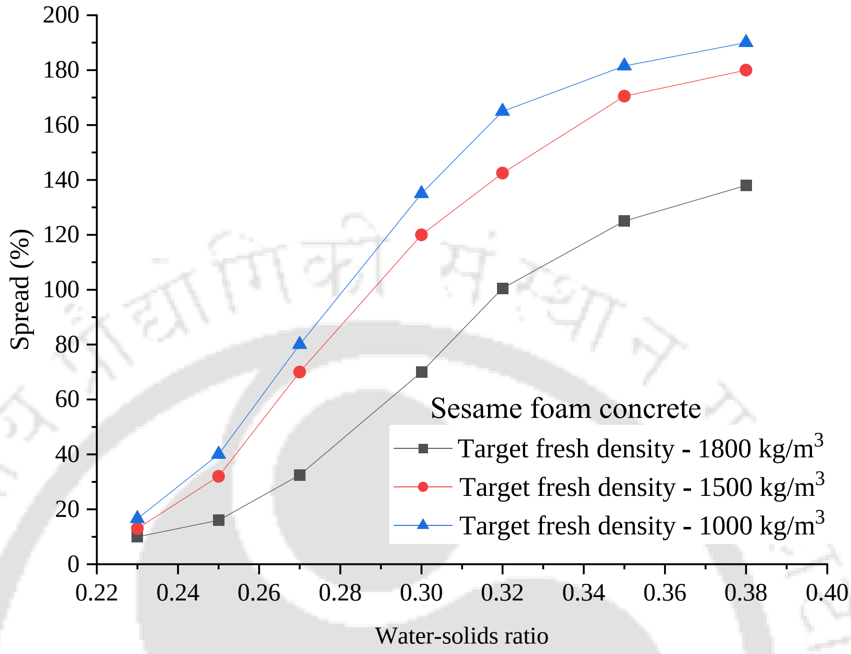
Table 5-3 Water-solids ratio requirement for production of stable foam concrete mixes

Target fresh density (kg/m^3)	Permitted range as per ASTM C869	Identified range of water-solids ratio for stable mixes	
		Sesame	Hingot
1000	1000 ± 50	0.25-0.32	0.27-0.32
1500	1500 ± 50	0.25-0.3	0.27-0.32
1800	1800 ± 50	0.23-027, 0.32	0.25-0.3

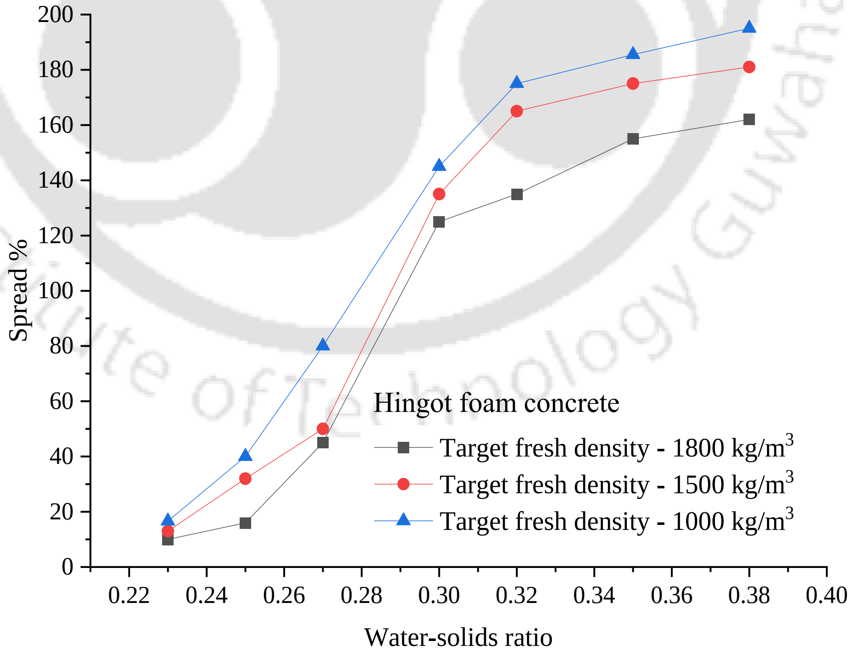
5.4.2 Effect of water-solids ratio on consistency of foam concrete mixes

The influence of water-solids ratio on the spread % for different foam concrete mixes are graphically presented in Figure 5-7. For the various mix compositions studied, inspection of obtained spread values indicate that increase in water-solids ratio from 0.23 to 0.38, results in a maximum increase of 93% in spread. Also, for a fixed water-solids ratio, increase in foam volume is found to result in increase in spread for all the foam concrete mixes studied indicating that foam volume has a greater influence on mix consistency. These findings contradict the observations of earlier researchers who proved that spread decreases with increase in foam volume as the foam addition increases the cohesiveness of the mix (Siva et al., 2017; Makul and Sua-Iam, 2016; Nambiar and Ramamurthy, 2008a). The reason for the deviation of experimental trend obtained in the present study can be attributed to the nature of foam (foam with higher foam density) used in the present study. The obtained trend of experimental observations made in the present study corroborates with the findings of researchers (Al-Shwaiter and Awang, 2021; Gencel et al., 2021; Ahmad and Chen, 2019; Sun et al., 2018; Chen et al., 2014; Chen and Liu, 2013; Panesar, 2013), who proved that the increase in air content resulted in increase in slump.

Further, comparison of the two surfactants indicate that hingot surfactant results in relatively higher spread when compared to that of sesame surfactant. For instance, for a water-solids ratio of 0.3, spread percentage observed for target density of 1800 kg/m^3 for hingot surfactant is 64% higher than that of sesame surfactant. The higher viscosity of the sesame surfactant solution contributes to the decrease in the spread percentage of sesame foam concrete mixes. This noticeable result and above critical remarks are fully in line with the findings of researchers who proved that use of high viscous surfactant solution affects the workability of foam concrete mix (Sahu and Gandhi, 2021; Yuanliang et al., 2021). Also, it is to be noted that the above difference in spread percentage between two different surfactants is found to significant only at water-solids of 0.3 and target density of 1800 kg/m^3. On contrary, such marked difference in spread % between two surfactants is not so evident at very low water- solids ratio of 0.25, indicating water content of the mix plays a major role than the viscosity of foam in relatively drier mixes. Also, it is logical to think that the spread test is not very sensitive to variation for relatively drier mixes. Furthermore, experimental evidence shows that the difference in spread percentage between two surfactants is less than 10% for mixes with higher foam volume (target density 1000 kg/m^3). Based on the experimental outcomes, the desired spreadability requirements to attain stable mixes ranges from 32 to 181.5% and 16 to 165% for surfactant sesame and hingot respectively. This is found to be comparable with the spread values reported in literature for foam concrete made with commercially available synthetic sodium lauryl sulphate surfactant (45 to 125%) (Sahu and Gandhi, 2021) and organic based natural surfactant (40 to 60%) (Nambiar and Ramamurthy, 2008a).



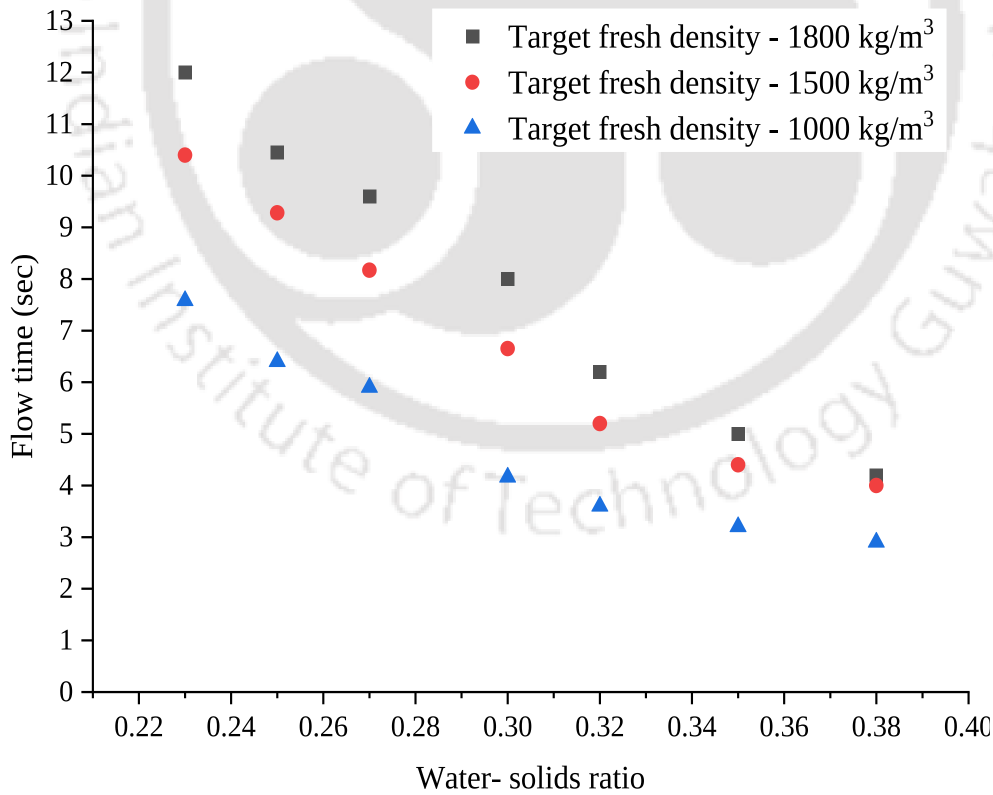
(a) Sesame foam concrete



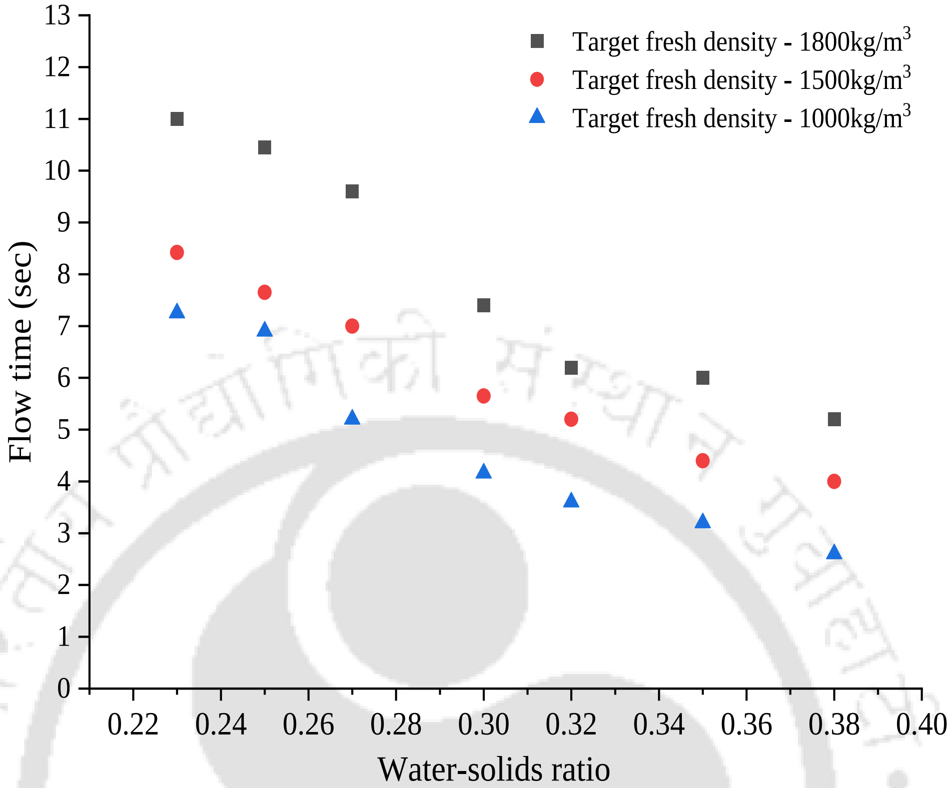
(b) Hingot foam concrete

Figure 5-7 Variation of spread percentage of foam concrete with water-solids ratio

Figure 5-8 shows the influence of water-solids ratio on flow time for various foam concrete mixes. The observations on flow time are found to follow the trend similar to that of spread %. Furthermore, the Marsh cone flow time studies provide a better picture of variability of consistency of foam concrete with surfactant type unlike the flow spread test, particularly for drier mixes with water-solids of 0.25. Experimental evidence shows that the spread percentage is similar for both the surfactants at low water-solids of 0.25. Contrary to above, the Marsh cone flow test represents a difference of 18% in flow time between hingot and sesame for the above case. On the other hand, for mixes with high water-solids ratio say 0.35, spread test shows difference in consistency of mixes with respect to variation in surfactant unlike the Marsh cone test. The flow time requirement to produce stable mix varies from 3.6 to 14.6 seconds and 4.16 to 10 seconds for sesame and hingot surfactants respectively for the different target densities studied.



(a) Sesame foam concrete



(a) Hingot foam concrete

Figure 5-8 Variation of flow time of foam concrete with water-solids ratio

5.4.3 Effect of water-solids ratio on air void characteristics of foam concrete

Air void size distribution

Figure 5-9 and 5-10 shows the sample frequency histogram and cumulative frequency distribution curves for air void size distribution of typical foam concrete mixes. The air void parameters D50 and D90 derived from cumulative frequency curves indicate medium air void size and 10% oversize air void respectively. Table 5-4 present the comparison of air void parameters of different concrete mixes. Firstly, the effect of variation of water-solids ratio (within the optimum water-solids ratio range) on the pore size distribution of sesame foam concrete will be discussed. The trend of variation of air void size distribution with water-solids ratio for a particular density of concrete is not very much clear from Figure 5-11(a). From Fig 5-12 (a), it can be observed that increase in water-solids ratio (within the range of water-solids ratio which results in stable mixes) results in increase in D90 (bigger size air voids) for a particular density of concrete (more evident in sesame foam concrete). Further, it is evident from Table 5-4, that D50 and D90 increases with increase in water-solids ratio of sesame foam concrete for all the target densities studied (within the optimum water-solids ratio range). A substantial increase of 35% in D90 is observed for increase in water-solids ratio from 0.25 to 0.32 for target density of 1000 kg/m^3 sesame foam concrete. The above observation is particularly noticeable in mixtures with higher foam volume as the chances for bubble coalescence is more in these mixes.

Concerning the variation of air void size distribution of hingot foam concrete with water-solids ratio (within the optimum water-solids ratio range), it is observed that the variation is rather small unlike sesame surfactant mixes (Table 5-4). Also, another very apparent thing is that the proportion of larger size air voids (>400 μm) is very much higher when compared to sesame surfactant mixes (Figure 5-11). The difference in the viscosities of two surfactant solutions contribute to the enormous difference in bigger size air voids. Further investigation of the impact of density of concrete on microstructure shows that D50 and D90 reduces with reduction in foam volume (Figure 5-12, 5-13 and 5-14). Compared to D50, D90 reduces sharply with reduction in foam volume. Furthermore, the air void parameter D50 obtained in present study is comparable with D50 value of 133 μm reported in literature for a similar mix with commercial synthetic SLS surfactant (Sahu and Gandhi, 2021). However, D50 air void sizes reported for natural animal protein foaming agent by Nambiar and Ramamurthy (2007a) is slightly on higher side of order of 325 μm for foam concrete with similar mix proportions and density of 1000 kg/m^3.

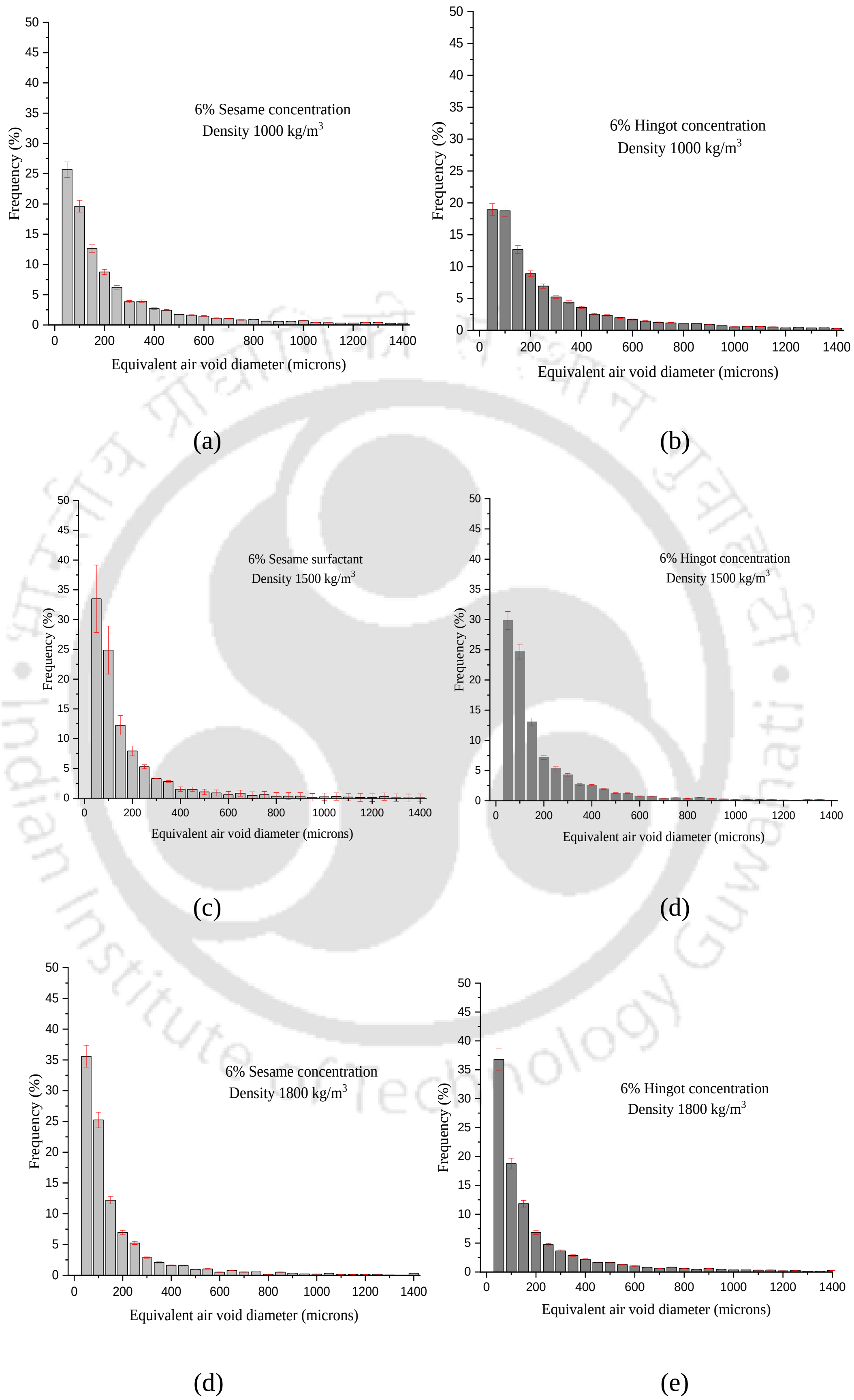


Figure 5-9 Frequency distribution of air void sizes in foam concrete

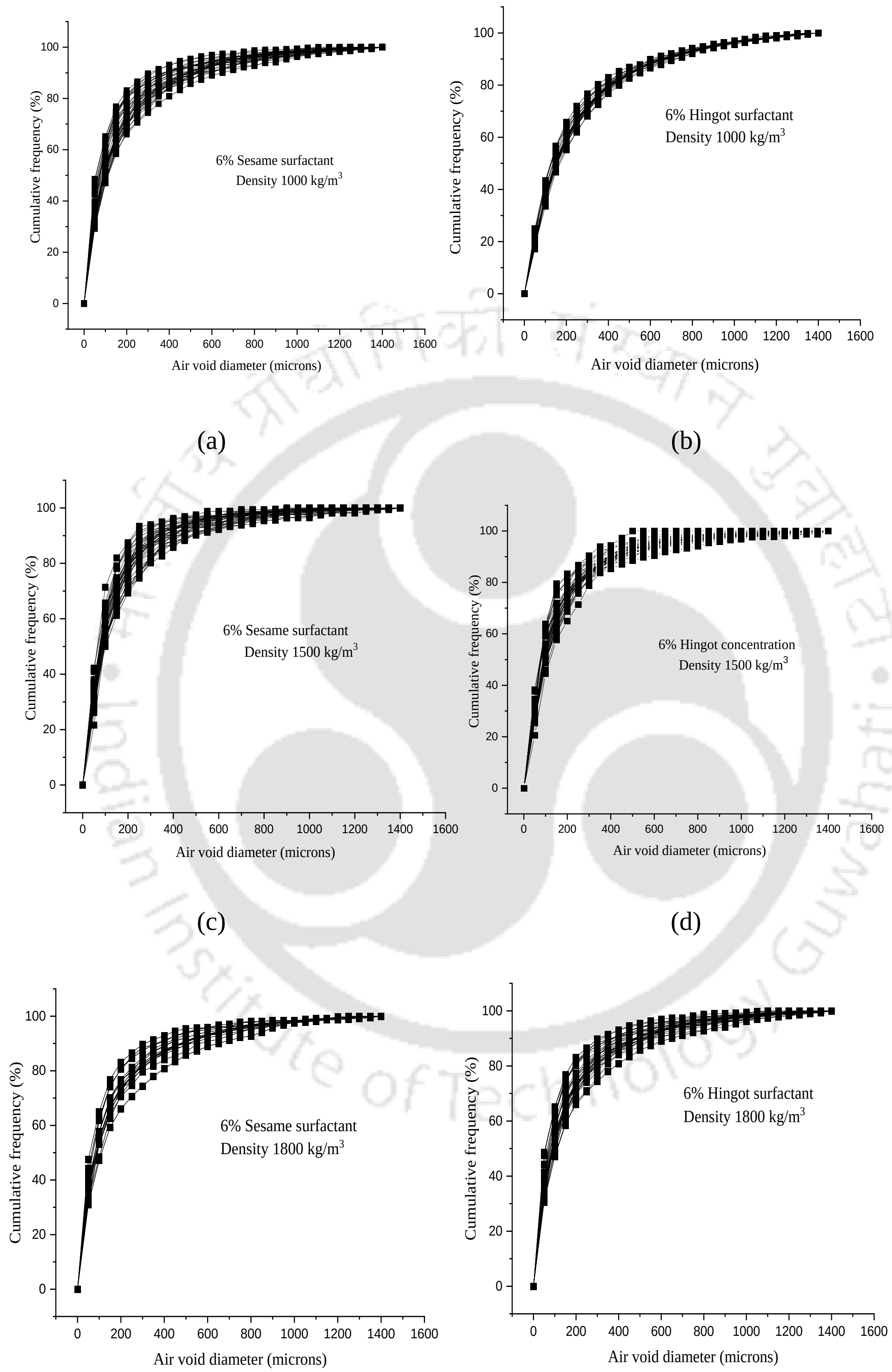


Figure 5-10 Cumulative frequency distribution of air void sizes in foam concrete

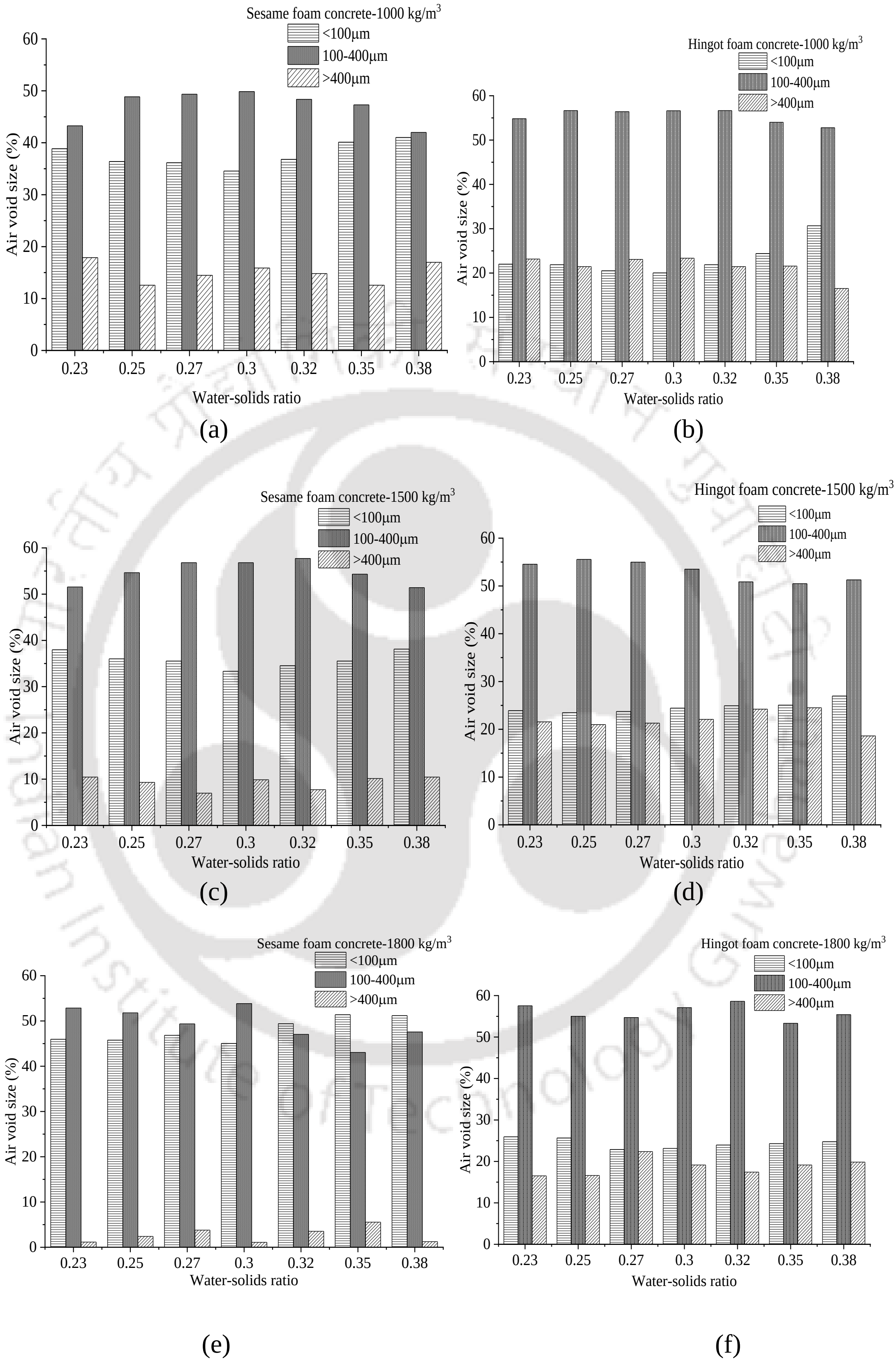
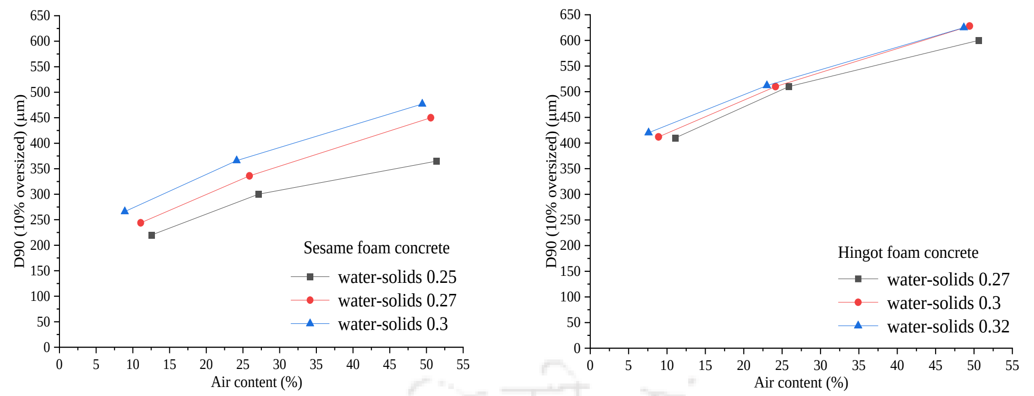
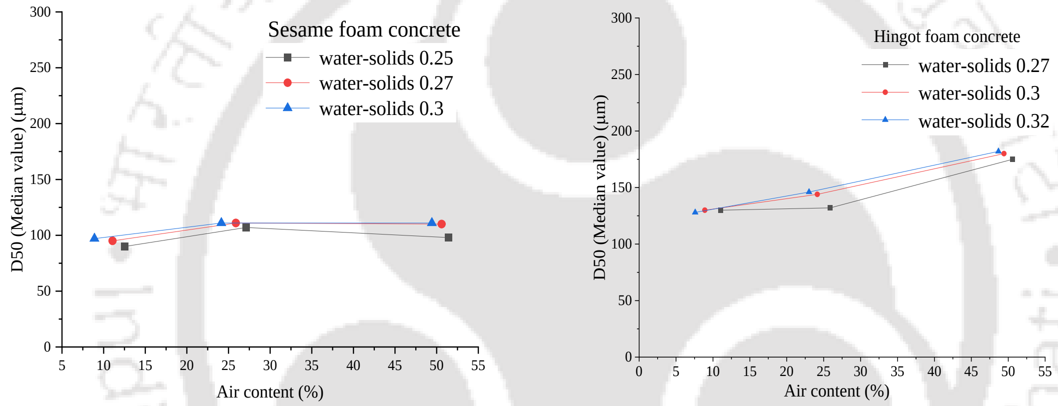


Figure 5-11 Effect of water-solid ratio on the pore diameter distribution



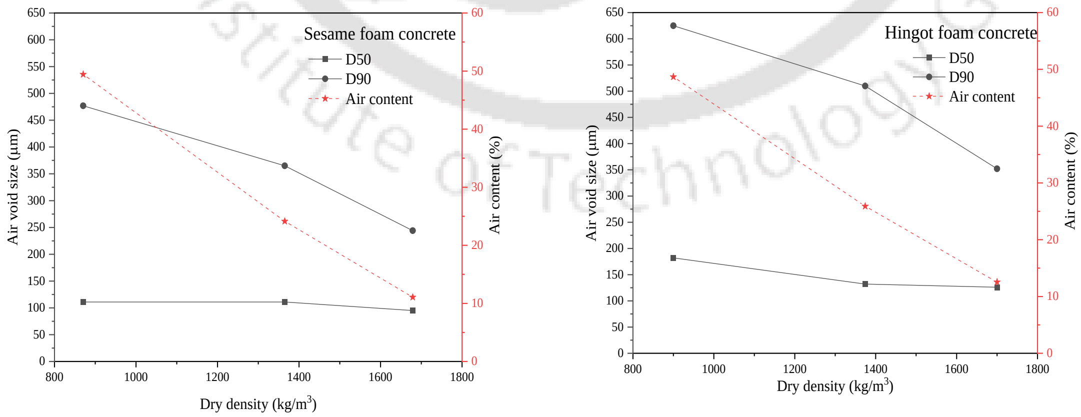
(a) (b)

Figure 5-12 Variation of D90 with air content



(a) (b)

Figure 5-13 Variation of D50 with air content



(a) (b)

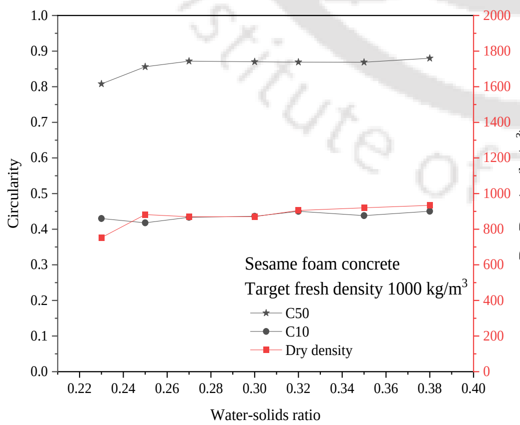
Figure 5-14 Variation of D50 and D90 with dry density and air void content

Table 5-4 Air void size distribution of foam concrete with different densities

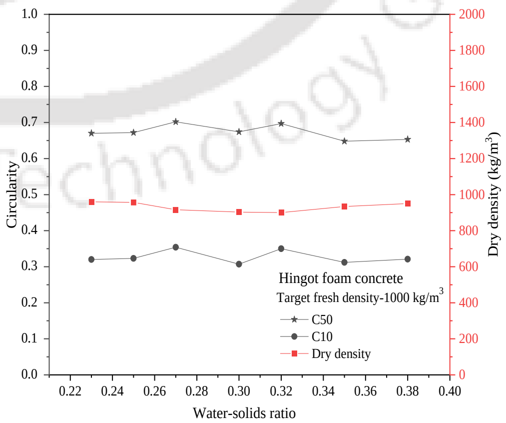
		Sesame		Hingot	
Density (kg/m^3)	**water-solids**	**D50 Average (Range)**	**D90 Average (Range)**	**D50 Average (Range)**	**D90 Average (Range)**
1000	0.23	103 µ (55-128)	400 µ (325-670)	195 µ (100-226)	688 µ (321-800)
1000	0.25	98 µ (52-124)	365 µ (304-650)	197µ (110-230)	700µ (342-870)
1000	0.27	110 µ (57-152)	450 µ (310-634)	175µ (110-230)	600µ (342-670)
1000	0.3	111 µ (59-150)	477 µ (318-697)	180 µ (112-250)	628 µ (350-664)
1000	0.32	114 µ (62-156)	492 µ (326-695)	182 µ (126-240)	625 µ (360-680)
1000	0.35	114 µ (58-160)	509 µ (355-758)	179 µ (120-261)	640 µ (380-780)
1000	0.38	117 µ (65-172)	525 µ (400-780)	170 µ (122-265)	676 µ (388-810)
1500	0.23	110 µ (84-146)	366 µ (210-550)	147 µ (100-208)	584 µ (260-650)
1500	0.25	107 µ (86-140)	300 µ (207-520)	140 µ (119-196)	550 µ (211-622)
1500	0.27	111 µ (98-149)	336 µ (220-535)	132 µ (106-125)	510 µ (215-525)
1500	0.3	111 µ (78-143)	366 µ (220-535)	142 µ (112-162)	512 µ (260-536)
1500	0.32	115 µ (80-150)	370 µ (232-606)	146 µ (112-198)	512µ (221-536)
1500	0.35	115 µ (91-177)	363 µ (230-612)	167 µ (110-222)	635 µ (331-771)
1500	0.38	116 µ (86-201)	378 µ (277-650)	170 µ (122-245)	650 µ (300-788)
1800	0.23	90 µ (50-123)	220 µ (176-400)	128 µ (74-160)	364 µ (200-420)
1800	0.25	90µ (58-129)	220 µ (180-401)	127 µ (116-240)	352µ (224-520)
1800	0.27	95 µ (57- 125)	244 µ (196-422)	130µ (112-162)	450 µ (260-508)
1800	0.3	97 µ (50-144)	266 µ (195-434)	130 µ (95- 160)	412 µ (216-425)
1800	0.32	100 µ (60-145)	270 µ (197-448)	128 µ (85-266)	420 µ (222-501)
1800	0.35	105 µ (58-166)	245 µ (195-450)	115 µ (101-265)	435 µ (235-550)
1800	0.38	98 µ (55-172)	299 µ (255-451)	120 µ (111-287)	442 µ (327-585)

Circularity of air voids

Figure 5-15 shows the variation in circularity of air void for different mixes. Results shows that there is no significant variation of circularity of air voids with respect to water-solids ratio. Another interesting thing to note is that there is a decrease in circularity with increase in density of sesame foam concrete. This is contradictory to the observations reported in most of the literature which states that circularity of air voids increases with reduction in foam volume. The above deviation can be ascribed to the presence of a higher proportion of smaller and irregular voids (<100µm), particularly in sesame foam concrete with target density of 1800 kg/m^3 (Figure 5-11 (c)). The above justification is supported by findings of Jiang et al. (2016) and Liu et al. (2016) who proved that very small pores are irregular and highly connected. Additionally, it is to be noted that the quantum of larger size air voids (>400µm) is lesser than 5% in the above mix. For hingot foam concrete mixes, in line with the trend reported in previous literature (She et al., 2018; Hilal et al., 2015a), the circularity values of foam concrete reduce with an increase in foam volume. The bubble coalescence in mixes with higher foam volume contributes to the reduction in circularity. Comparison studies on two different types of surfactants, indicate that sesame surfactant solution which is relatively more viscous than hingot shows higher circularity air voids which is particularly noticeable in mixtures with higher foam volume.



(a)



(b)

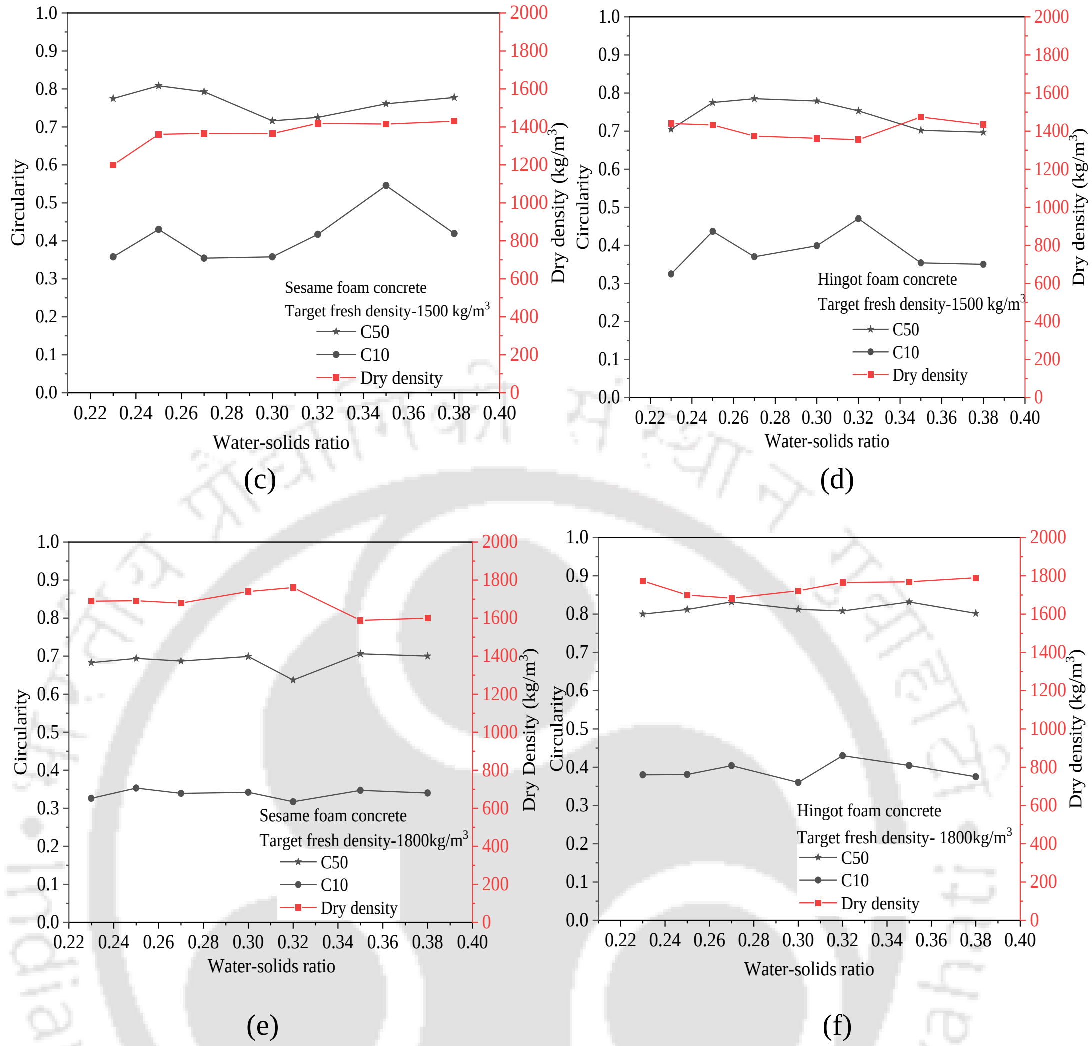


Figure 5-15 Variation of circularity with respect to water solid ratio and dry density

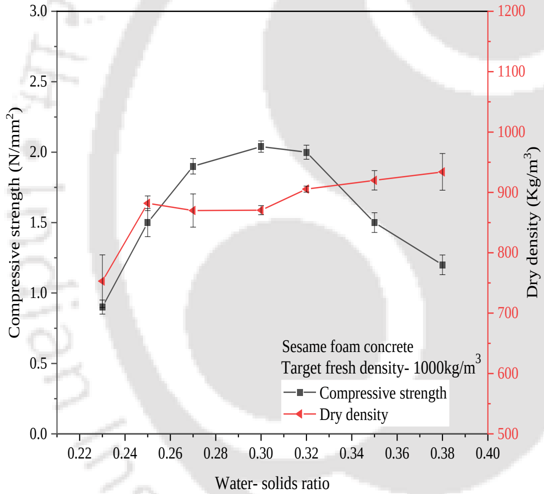
5.4.4 Effect of water-solids on mechanical behaviour of foam concrete

This section reports the findings of research on the impact of water-solids ratio, foam volume, and surfactant type on the compressive strength and tensile strength of foam concrete. The relationship between density, strength, and air void characteristics will also be explored, along with the experimental results rationale.

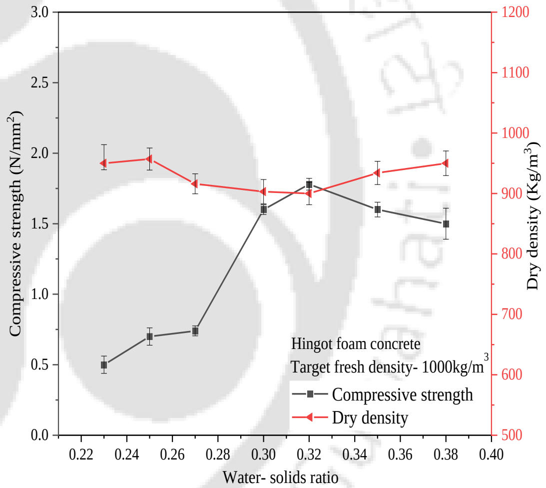
The implication of water-solids ratio on compressive strength of sesame foam concrete for different target fresh densities is apparent in Figure 5-16. Considering the range of water-solids ratio which results in stable mixes, it is evident that for most of the mixes studied, the compressive strength of foam concrete increases with water-solids ratio first and then decreases. The increase in compressive strength with increase in water-solids ratio till the optimum point can be attributed to the greater amount of hydration of cement at higher water content. These experimental outcomes agree with the available literature (Piotrowski and Prochoń, 2018; Falliano et al., 2018a; Liu et al., 2013; Yang and Lee, 2013). For the present study, increase in water-solids ratio from 0.25 to 0.3 results in 5.3% reduction of foam content requirement for a given target density of 1000 kg/m^3 which again contributes to the increase in strength. On the other hand, use of water-solids ratio higher than the optimum, results in larger pore size distribution range which can be attributed to the lesser strength.

When the compressive strength of mixes whose density lies outside the desired target range are compared with stable mixes, it is evident that at both extreme points of water-solids ratio (say 0.23 and 0.38), the strength is very much lower than at the optimum water-solids of 0.3. The trend of variation of air void size distribution with water-solids ratio for a particular density of concrete is not very much clear from Figure 5-11(a). From Figure 5-12 (a), it can be observed that increase in water-solids ratio (within the range of water-solids ratio which results in stable mixes) results in increase in D90 (bigger size air voids) for a particular density of concrete (more evident in sesame foam concrete). Table 5-4 also supports the above observations indicating that there is overall increase in D90 (bigger size air voids) with increase in water-solids ratio which can be attributed to variation in strength.

Table 5-5 shows the optimum water-solids ratio derived based on the maximum compressive strength for foam concrete with different target densities. Comparison of compressive strength of foam concrete mixes at optimum water-solids ratio for target density of 1800 kg/m^3 indicates that compressive strength of sesame foam concrete is 34% higher than that of hingot foam concrete. The above observation on strength corroborates with the experimental results of air void size distribution which indicates that D90 air void is 84% higher in hingot than sesame for the similar mix. Figure 5-17 shows that increasing the diameter of the air void leads to a decrease in strength, implying that the diameter and strength are inversely related. Further the higher viscous nature of sesame surfactant can be attributed to the improved microstructure and higher strength of sesame foam concrete. The variation of D50 with strength is similar for both the surfactants as the variation of D50 is narrower. Further, comparison with foam concrete mixes of similar mix proportion and design density of 1000 kg/m^3 made with commercially available synthetic surfactant SLS (as reported by Sahu and Gandhi (2021)) shows that mixes adopted in the present study exhibits 30 to 57% higher compressive strength. On a similar note, foam samples made in present study is found to outperform compressive strength of foam concrete made with other organic based surfactant (Nambiar and Ramamurthy, 2006) by 38 to 66%. The smaller air void size microstructure of the mixes produced in present study can be attributed to the relatively higher compressive strength. In this line, foam concrete of design density 1000 kg/m^3 made with commercial Neopor foaming agent (animal protein based) and mineral admixtures such as fly ash has shown relatively higher compressive strength of 3.2 MPa (Bartenjeva and Mashkin, 2017). The above data highlights the scope for further improvement of mixes adopted in the present study through incorporation of different admixtures.



(a)



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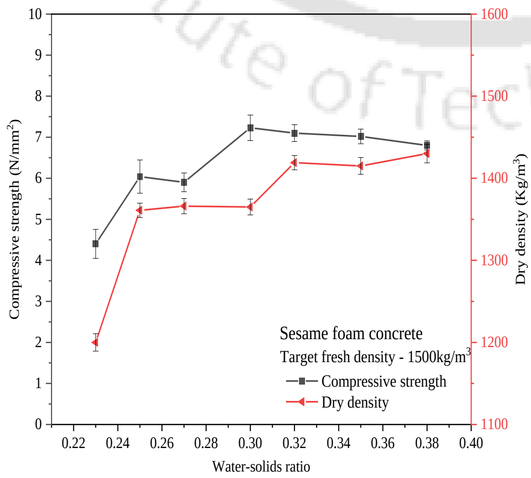

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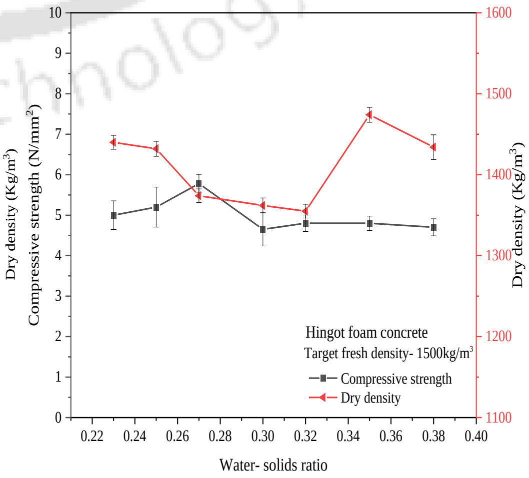

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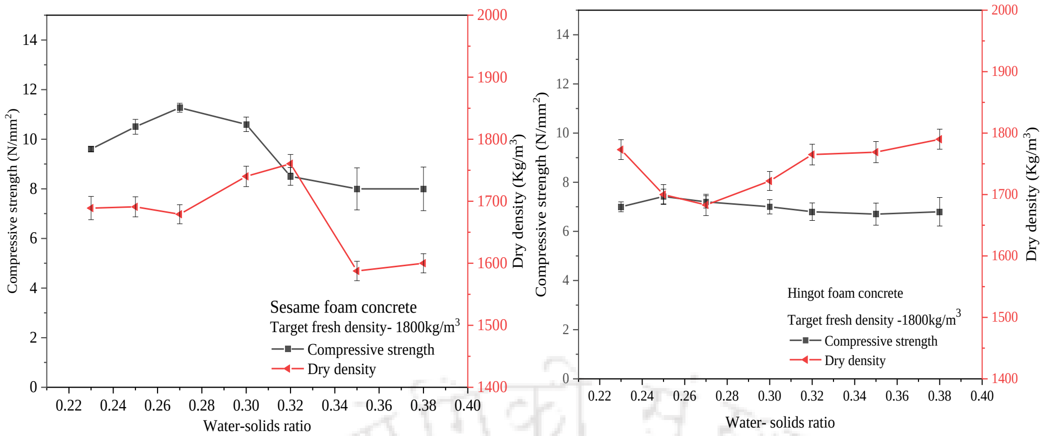
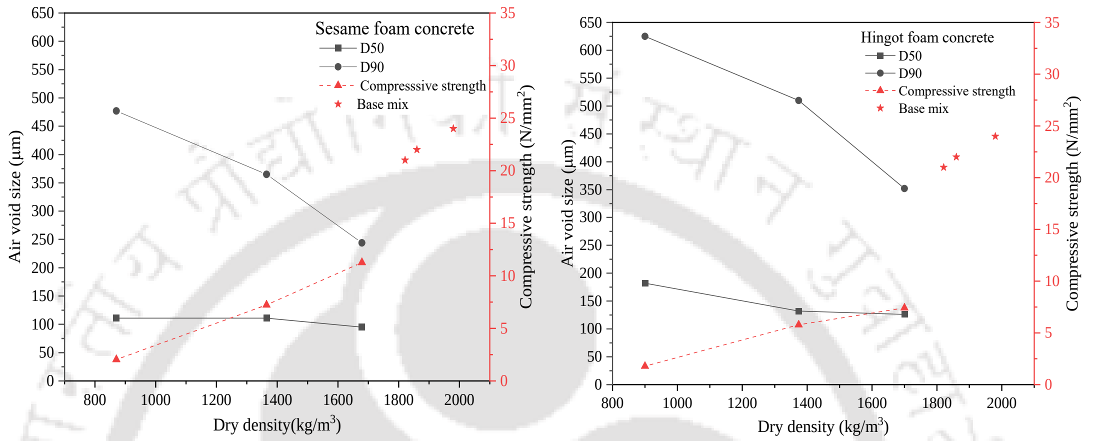


(e) (f)

Figure 5-16 Variation of compressive strength with respect to water-solids ratio

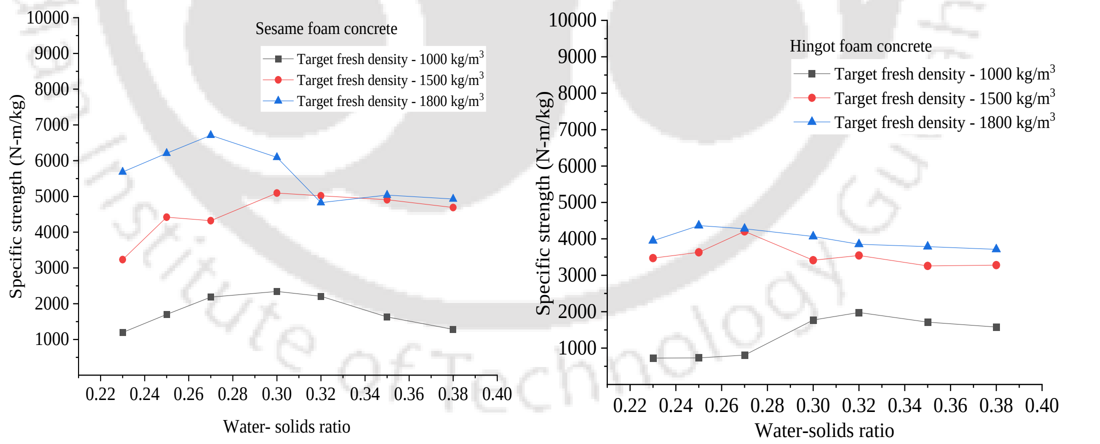
Also, the Figure 5-17 indicates that the compressive strength of concrete has a direct relationship with density, supporting the hypothesis that density plays an important role in determining the strength (Ramamurthy et al., 2009; Kearsley and Wainwright, 2001c). Obviously, the base mixes (mortar without foam) being denser than the comparable foam concrete mixes, possess higher compressive strength. Specific strength (strength/density) is also studied to eliminate the effect of variation in density on strength. Figure 5-18 and 5-19 indicate that both the specific strength and split tensile strength follow a trend similar to that of compressive strength, showing maximum strength at optimum water-solids ratio.

In addition, rough material cost analysis information is presented; it is discovered that the cost of sesame seed and hingot fruit for one cubic metre of foam concrete production (target density 1000 kg/m^3) are ₹629 and ₹105 respectively. The above mentioned cost figures of natural surfactants, particularly hingot is very much economically beneficial when compared on similar basis with commercially available surfactants such as synthetic based SLS (₹896) (Sahu and Gandhi, 2021) and animal protein based Neopor (₹1413) (Bartenjeva and Mashkin, 2017) in market. Further, currently research is being undertaken to prove that the associated environmental loads for production of aforementioned natural surfactants is minimal when compared to commercially used synthetic surfactants. Furthermore, the approximate material cost estimates reveal that the price of one cubic metre of foam concrete with a target density of 1000 kg/m^3 at the optimal surfactant concentration is ₹2706 and ₹2149 for the surfactants sesame and hingot, respectively which is quite nominal. The corresponding cost data for foam concrete with synthetic based SLS and animal protein based Neopor are ₹2972 and ₹3458 respectively. Hence, the natural surfactants used in this study has great potential to replace the synthetic surfactants from economic and environment perspectives.



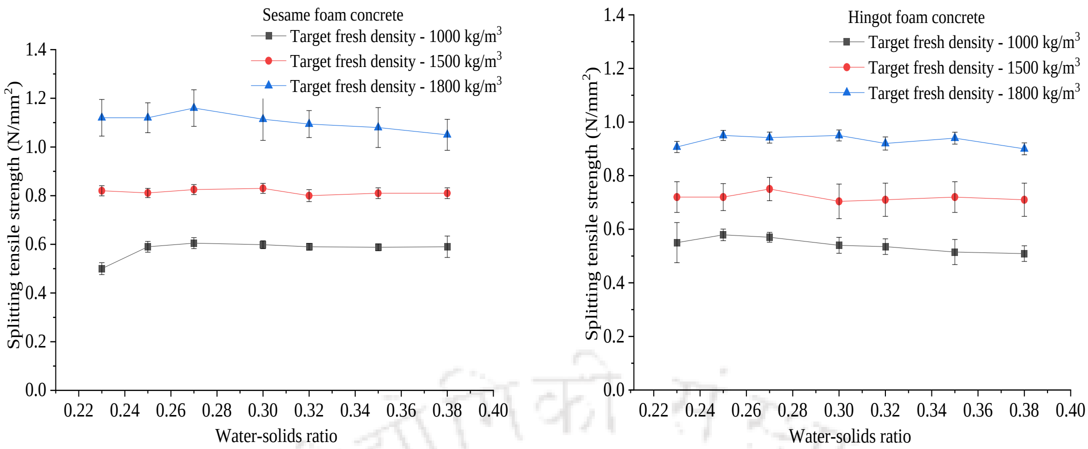
(a) Sesame foam concrete (b) Hingot foam concrete

Figure 5-17 Correlation of compressive strength, air void size and dry density



(a) Sesame foam concrete (b) Hingot foam concrete

Figure 5-18 Variation of specific strength with water-solids ratio



(a) Sesame foam concrete (b) Hingot foam concrete

Figure 5-19 Variation of tensile strength with water-solids ratio

Table 5-5 Air-void system and mechanical properties of foam concretes with different water-solids ratios

Mix	**Wet design (kg/m^3)**	**Dry density (kg/m^3)**	**Design air content (%)**	**Air void D50 (µm)**	**D90 (µm)**	**fc/fcu_b**	**ft/fc**	**Specific strength**
Target density- 1000 kg/m^3-sesame								
0.23	800	753	52.24	103	517	0.0501	0.5555	0.00119
0.25	1006	882	51.4	98	365	0.0838	0.3933	0.00170
0.27	985	870	50.59	110	450	0.1092	0.3184	0.00218
0.3	995	870.5	49.43	111	477	0.1189	0.2933	0.00234
0.32	1030	905.4	48.68	114	492	0.1167	0.295	0.00220
0.35	1040	920	47.6	114	509	0.0875	0.392	0.00163
0.38	1050	934	46.56	117	525	0.0706	0.4916	0.00128
Target density- 1500 kg/m^3-sesame								
0.23	1200	1200	28.36	110	366	0.2449	0.1636	0.00323
0.25	1454.94	1361	27.11	107	300	0.3378	0.1192	0.00442
0.27	1515	1366	25.89	111	336	0.3391	0.1271	0.00432
0.3	1516.5	1365	24.14	111	365	0.4213	0.0973	0.00509
0.32	1575	1419	23.02	115	370	0.4142	0.1	0.00501

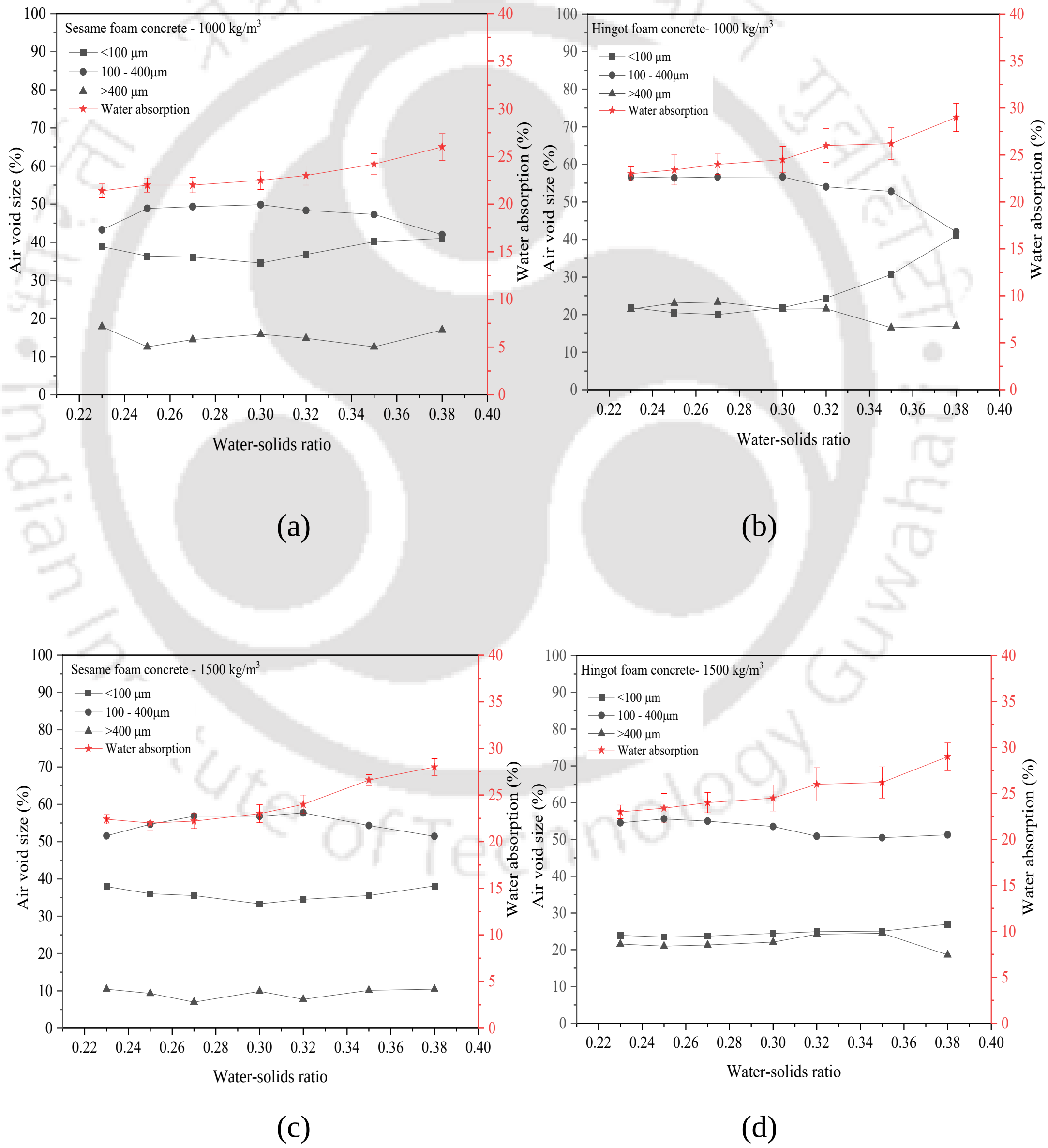
Mix	Wet design (kg/m³)	Dry density (kg/m³)	Design air content (%)	Air void D50 (μm)	Air void D90 (μm)	fc/fcu_b	ft/fc	Specific strength
0.35	1552.5	1415	21.4	115	363	0.4093	0.1025	0.00491
0.38	1575	1430	19.84	116	378	0.4	0.1044	0.00468
Target density- 1800 kg/m³-sesame								
0.23	1818	1689	14.04	90	220	0.4633	0.1166	0.00568
0.25	1827	1691	12.53	90	220	0.5121	0.1066	0.00621
0.27	1796.4	1679	11.07	95	244	0.5497	0.1029	0.00671
0.3	1818	1740	8.9	97	266	0.5190	0.1051	0.00601
0.32	1836	1760.4	7.6	100	270	0.425	0.1287	0.00482
0.35	1728	1587.6	5.6	105	245	0.4073	0.135	0.00503
0.38	1719	1558.2	3.8	98	299	0.33	0.1312	0.00492
Target density- 1000 kg/m³-hingot								
0.23	1080	960	52.24	195	688	0.0238	1.1	0.000700
0.25	1070	957	51.40	197	700	0.0392	0.8271	0.000731
0.27	1025	916	50.6	175	600	0.0426	0.770	0.000807
0.3	1010	903	49.43	180	628	0.0933	0.3375	0.001772
0.32	1020	900	48.68	182	625	0.1039	0.3005	0.001978
0.35	1050	934	47.6	179	640	0.0933	0.3218	0.001713
0.38	1080	950	46.56	169	676	0.0883	0.3393	0.001578
Target density- 1500 kg/m³-hingot								
0.23	1590	1440	28.36	147	584	0.2439	0.164	0.003472
0.25	1560	1432	27.11	140	550	0.26	0.1559	0.003631
0.27	1515	1374	25.89	132	510	0.2948	0.1427	0.004206
0.3	1489.5	1362	24.14	144	512	0.2325	0.1784	0.003414
0.32	1455	1355	23.02	146	512	0.2526	0.1666	0.003542
0.35	1590	1474	21.4	167	615	0.2341	0.16875	0.003256
0.38	1575	1434	19.84	170	650	0.2410	0.1723	0.003277
Target density-1800 kg/m³ -hingot								

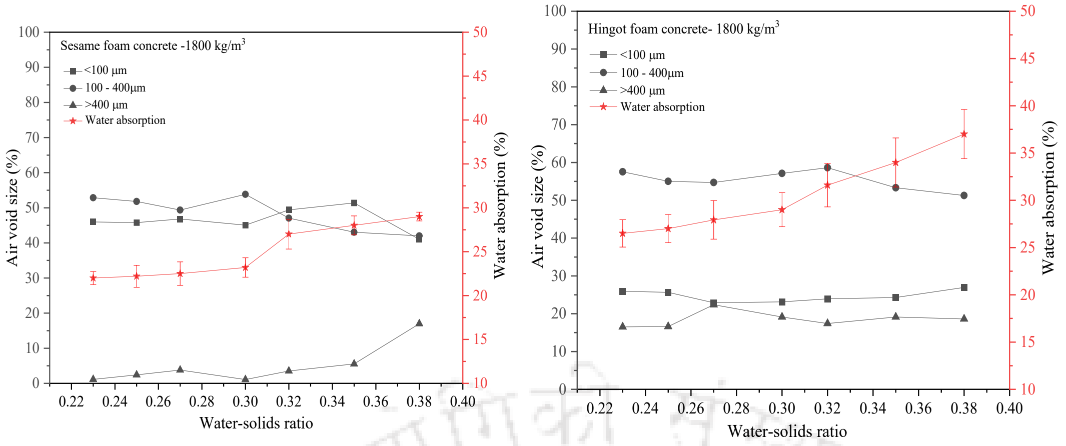
Mix	Wet design (kg/m³)	Dry density (kg/m³)	Design air content (%)	Air void D50 (µm)	Air void D90 (µm)	fc/fcu$_b$	ft/fc	Specific strength
0.23	1890	1773	14.04	128	364	0.3378	0.1295	0.003948
0.25	1818	1700	12.53	126	352	0.3619	0.1280	0.004365
0.27	1773	1683	11.07	130	410	0.3512	0.1308	0.004278
0.3	1836	1722	8.97	130	412	0.3428	0.1357	0.004065
0.32	1872	1765	7.6	128	420	0.34	0.1352	0.003853
0.35	1890	1769	5.6	115	435	0.3411	0.1402	0.003788
0.38	1906	1766	3.8	120	442	0.2833	0.1323	0.003770

fc- compressive strength of foam concrete; fcu$_b$ – compressive strength of base mix, ft- tensile strength

5.4.5 Effect of water-solids ratio on water absorption

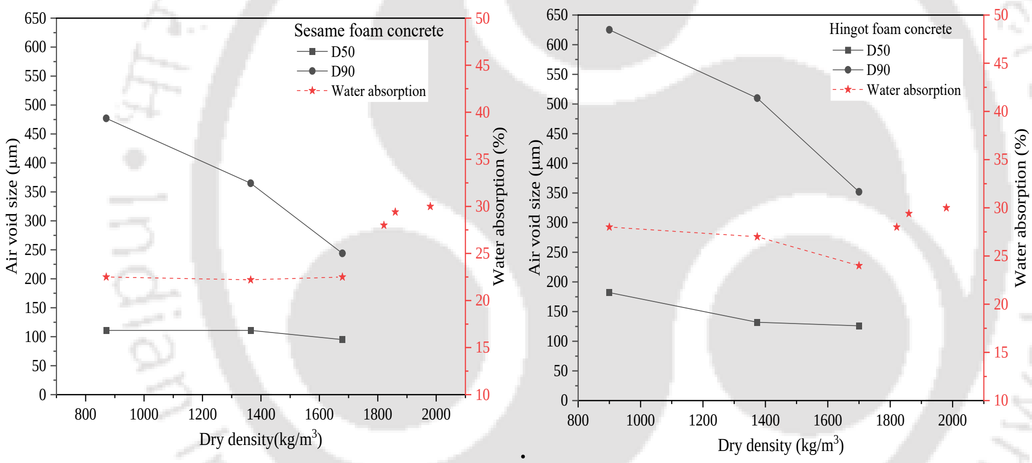
Figure 5-20 shows that the variation of water absorption with water-solids ratio (within stable range) is not significant for all the mixes studied. However, when the water absorption of unstable mixes is compared with stable mix, it is observed that at extreme unstable point of water-solids ratio (say 0.38), the water absorption is found to be 15-25% higher than at stable water-solids ratio of 0.3. This can be attributed to significant increase in smaller and connected capillary pores in mixes with higher water-solids ratio as discussed by Jiang et al. (2016). In this line, the air void analysis results in the present study show a significant increase of 19 to 53 % of smaller pores (<100µm) at a higher water-solids ratio of 0.38 particularly for lower density foam concrete mixes for both the surfactants (Figure 5-20 (a) and (b)). On the other hand, in 1800 kg/m^3 sesame foam concrete increase in water absorption at water-solids of 0.38 can be attributed to significant increase of bigger size pores (>400µm) (Figure 5-20 (c)). Following that, the variation of water absorption of foam concrete with density of concrete indicates that 16% increase in water absorption is observed with increase in foam volume for hingot foam concrete when compared at optimum water-solids ratio. The increase in water absorption can be attributed to more connected pore structure in mixes with high foam volume as reported by other researchers (Jiang et al., 2016; Liu et al., 2016). However, the variation in water absorption with foam volume is not significant for sesame foam concrete mixes (Figure 5-21). Comparison of water absorption of two surfactants indicate that water absorption of hingot foam concrete is 22% higher than that of sesame foam concrete and the above difference is more apparent in mixes with higher foam volume. The higher proportion of bigger pores (>400 µm) in hingot can be ascribed to higher water absorption of hingot foam concrete. Further, the obtained results of water absorption are comparable with results reported for synthetic SLS surfactant (Sahu and Gandhi, 2021) and organic foaming agent (Nambiar and Ramamurthy, 2007b) for concrete mixes with similar mix proportions.





(e) (f)

Figure 5-20 Correlation of water absorption and air void size with variation in water-solids ratio



(a) (b)

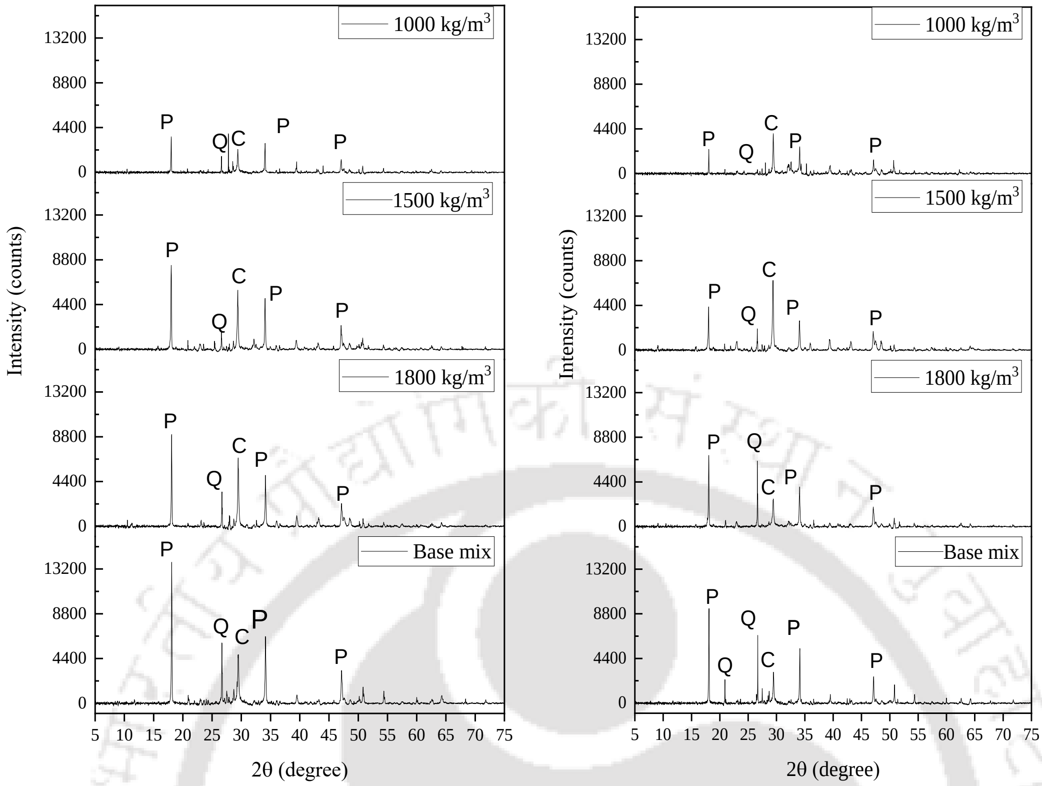
Figure 5-21 Correlation of water absorption, air void size and dry density

5.4.6 Mineralogical analysis

To elucidate the reasons for various behaviour, as a next step, the microstructural investigations of foam concrete produced with two different natural surfactants was performed using XRD and EDS. Diffraction patterns of the sesame foam concrete samples and hingot foam concrete samples of different target densities produced at their optimum water-solids ratios (after 28 days of curing) along with the base mixes are presented in Figure 5-22.

The peaks of the following primary phases namely Quartz (2θ values: 20.85, 26.652, 55.328, 68.323 and 77.662, Reference Code: 00-046-1045), Portlandite (18.008, 47.121 and 77.654, Reference Code: 00-044-1481) and Calcite (2θ values: 29.0, 48.58 and 56.558, Reference Code: 00-005-0586) are identified in the XRD pattern of all samples. In base mix samples, the amplitude of the portlandite peaks and quartz peaks are noticeably higher than the foam concrete mixes (Figure 5-22). It can be assumed that, greater volume of paste and higher degree of hydration of base mix can be the reason for higher intensity of peaks of quartz and portlandite. Further XRD comparisons of different densities of foam concrete also indicates a similar trend with greater amplitude of quartz and portlandite in higher density (1800 kg/m^3) foam concrete mixes in comparison to lower density foam concrete mix (1000 kg/m^3). In support of earlier observations, studies conducted by Chung et al., (2020) and Sahu and Gandhi (2021) have proved that mixes with greater degree of hydration exhibited greater amplitude of portlandite.

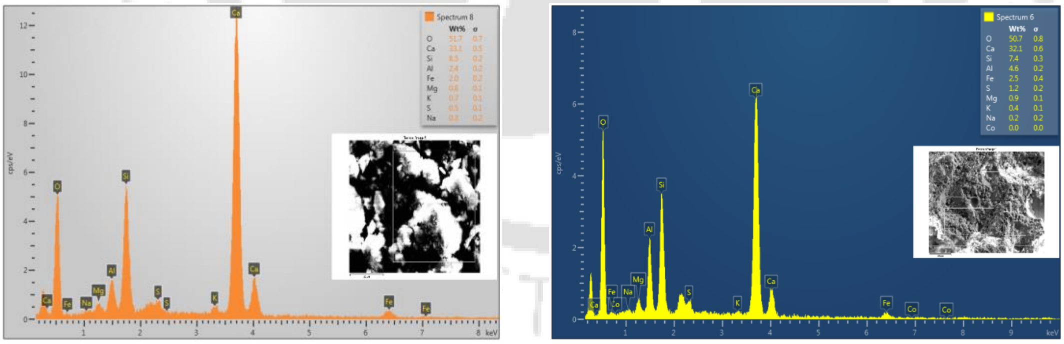
Furthermore, similar to study conducted by (Kunther et al., 2017) the results of EDS analysis on Ca/Si ratio can be used to justify the mechanical performance of concrete. Figure 5-23 represents the EDS spectrum of sesame and hingot foam concrete samples of different target densities along with their base mixes. According to EDS measurement, comparisons of Ca/Si ratio indicate that base mixes with relatively higher compressive strength than foam concrete mixes show relatively lower Ca/Si ratio. On a similar note, Ca/Si ratio of foam concrete with target density 1800 kg/m^3 is lesser than that of foam concrete with target density 1000 kg/m^3 (Table 5-6). Furthermore, comparison of sesame and hingot mixes indicate that sesame foam concrete mixes have relatively lower Ca/Si ratio as justified by its higher strength.



P: Portlandite, Q: Quartz, C: Calcite

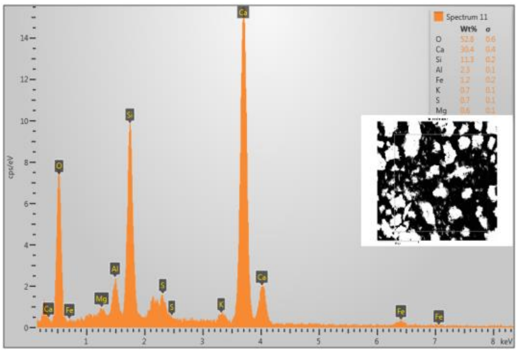
(a) Sesame foam concrete (b) Hingot foam concrete

Figure 5-22 XRD spectra of different mixes

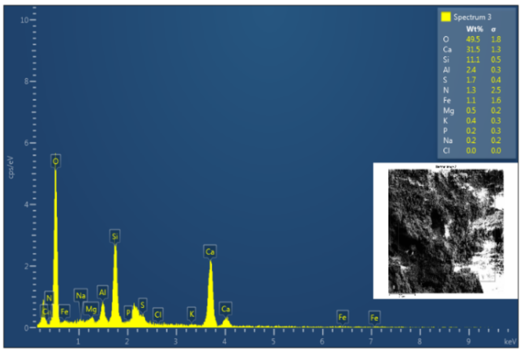


(a) Sesame foam concrete - Target density- 1000 kg/m^3

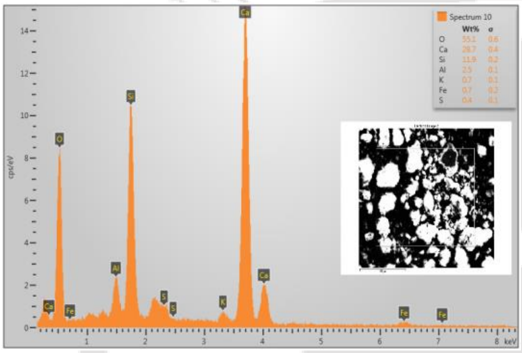
(b) Hingot foam concrete- Target density- 1000 kg/m^3



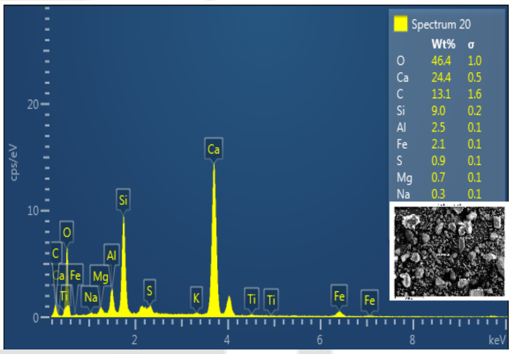
(c) Sesame foam concrete – Target density- 1500 kg/m^3



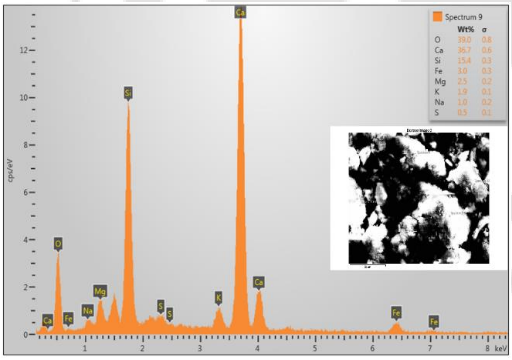
(d) Hingot foam concrete- Target density- 1500kg/m^3



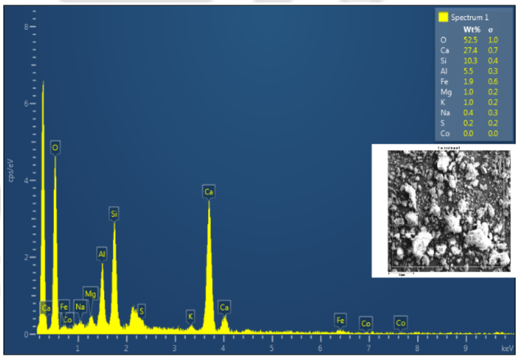
(e) Sesame foam concrete – Target density- 1800 kg/m^3



(f) Hingot foam concrete- Target density- 1800 kg/m^3



(g) Sesame foam concrete – Base mix



(h) Hingot foam concrete – Base mix

Figure 5-23 EDS spectrum of foam concrete

Table 5-6 Ca/Si ratio of foam concrete mixes with optimum water-solids ratio

Design density (kg/m^3)	**Ca/Si**	
	Sesame	**Hingot**
1000	3.89	4.33
1500	2.69	2.83
1800	2.41	2.7
Base mix	2.38	2.66

5.5 Summary

In this study foam concrete is produced with two different natural surfactants extracted from protein rich sesame and saponin rich hingot. The potential of use of these two surfactants in foam concrete and the role of water-solid ratio on various physical, chemical and mechanical properties of concrete have been considered for range of densities. It has been found that performance of foam concrete with different foaming agents varies greatly due to creation of different void structure in concrete. Comparative studies on consistency of foam concrete produced with two different surfactants indicate that the consistency of hingot foam concrete mix is relatively higher due to relatively less viscous nature of hingot surfactant when compared to that of sesame. On a similar note, in comparison to sesame surfactant, hingot surfactant foam concrete mixes exhibit bigger size air voids. Further, the experimental outcomes proved that the mechanical behaviour of porous material depends on both porosity and pore size distribution. The compressive strength and water absorption of sesame foam concrete with target density 1800 kg/m^3 is 56% higher and 7% lower respectively than that of hingot foam concrete. The above results corroborate the air void characterization results, which proved bigger size air voids in hingot foam concrete. Similar to observations reported in relevant literature, it is found that there is an optimum water-solids for a particular density of foam concrete at which the strength is maximum. Further the optimized water-solids ratio is derived for various densities and justified with air void structures. In line with the results mixes with optimum water-solids ratio are found to exhibit minimum water absorption. The results of mineralogical analysis proved that mixes with higher strength showed higher peaks of Portlandite and lower Ca/Si ratio indicating greater amount of hydration.

Chapter 6

Conclusions and Scope for Future Work

6.1 Conclusions

The salient conclusions arising out of this research work are summarised in the following two sections.

(i) Potential of Balanites aegyptiaca and Sesamum indicum for surfactant preparation and application in foam concrete.

(ii) Effect of type of foaming agent and water to solid ratio on foam concrete behaviour.

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

Potential of *Balanites aegyptiaca* and *Sesamum indicum* for surfactant preparation and application in foam concrete.

- ✓ General comparison between two surfactants shows that sesame surfactant is highly viscous when compared to hingot. Further, the variation of main effects of independent variables such as surfactant concentration, heating temperature and heating duration have substantial effect on the viscosity of sesame surfactant.
- ✓ For compressed air foam, higher level of foam generation pressure is needed for thorough mixing of air and liquid which eventually results in good quality foam with reduced liquid fraction, slower drainage rate and smaller size foam bubble microstructure.
- ✓ Surfactant concentration has significant effect on all the responses studied. Further the hydrolysis of protein and saponin based surfactant is very much dependent on the heating process adopted for surfactant extraction. Experimental outcomes indicate that increase in surfactant concentration, heating temperature and heating duration results in increase in viscosity of surfactant solution subsequently leading to reduction in liquid fraction, foam drainage and bubble size.

- ✓ Good correlation is established between viscosity of surfactant solution, foam density, foam drainage and foam bubble size.
- ✓ From the foam stability test in cement paste it is observed that both the surfactants are suitable for use in foam concrete production when optimized foam production parameters are adopted.

Effect of type of foaming agent and water to solid ratio on foam concrete behaviour.

- ✓ Comparative studies on consistency of foam concrete with two different surfactants indicates that the spread and flow time of hingot foam concrete mixes are relatively higher and lower respectively than that of sesame foam concrete mix for most of the mixes studied. The enormous difference observed here can be attributed to relatively lesser viscous nature of hingot surfactant solution when compared to sesame surfactant.
- ✓ Experimental studies on air void characterization reveal that a significant increase of 35 % in D90 (bigger size air voids) is observed with an increase in water-solids from 0.25 to 0.32 for sesame foam concrete with target density 1000 kg/m^3. In addition, closer examination of pore size distribution highlights that in comparison to sesame surfactant, hingot surfactant foam concrete mixes exhibit bigger size air voids, which can be ascribed to lesser viscosity of hingot surfactant solution.
- ✓ The experimental outcomes proved that the mechanical behaviour of porous material depends on both porosity and pore size distribution. It is found that there is an optimum water-solids for a particular density of foam concrete at which the strength is maximum. The optimized water-solids ratio derived for various mixes was justified with air void structure. In line with earlier findings, mixes with optimum water-solids are found to exhibit minimum water absorption.
- ✓ The compressive strength and water absorption of sesame foam concrete with target density 1800 kg/m^3 is 56 % higher and 7 % lower respectively than that of hingot foam concrete. The above results corroborate the air void characterization results, which proved bigger size air voids in hingot foam concrete.

✓ The results of mineralogical analysis proved that mixes with higher strength showed higher peaks of Portlandite and lower Ca/Si ratio indicating greater amount of hydration.

6.2 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) Investigations on additives to improve the quality of foam can be carried out.

ii) Identification of more compatible chemical admixtures to reduce the water-solid ratio requirement is needed

iii) Studies on use of mineral admixtures such as fly ash and slag can be carried out to improve the mechanical and durability related performance of foam concrete.

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

v) Environmental impact of foam concrete with identified natural surfactants could be explored.

vi) Studies on prototype constructed with foam concrete blocks and panels can facilitate faster commercialization of identified natural surfactant.

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Publications/ Conference/ Symposium based on Research

Publications

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. **Khwairakpam, S.,** Gandhi, I. S. R., and Sahu, S. S. (2020). "***State-of-the-art review on shrinkage behaviour of foamed concrete".*** *Journal of Structural Engineering* 46 (6), 475-484.
3. **Khwairakpam, S.,** and Gandhi, I. S. R. (2020). ***"Assessment of the potential of a naturally available foaming agent for use in the production of foam concrete".*** *Materials Today: Proceedings,* 32, 896-903.
4. **Khwairakpam, S.,** and Gandhi, I. S. R. (2021). ***"Development of Natural Based Foaming Agent for the Potential Use in Foam Concrete".*** *Indian Concrete Journal,* 95 (7), 7-13.
5. **Khwairakpam, S.,** and Gandhi, I. S. R. (2022). "***Comprehensive investigation into the effect of the newly developed natural foaming agents and water to solids ratio on foam concrete behaviour".*** *Journal of Building Engineering, 58,* 105042
6. **Khwairakpam, S.,** and Gandhi, I. S. R. "***Novel investigation on potential of Balanites aegyptiaca and Sesamum indicum for surfactant preparation and application in foam concrete".*** Journal of Materials in Civil Engineering (with Editor)

Conference/Symposium

1. **Khwairakpam, S.,** Sahu, S. S., and Gandhi, I. S. R. "State-of-the-Art Review on Potential of Different Sources of Natural Surfactant and Methods of Preparation of Surfactant for Use in Foam Concrete Production." in Research

Scholar Symposium - Concrete Research in India (CRI) on 13th December (2018), organized by IIT Bombay, Mumbai.

2. **Khwairakpam, S.,** and Gandhi, I. S. R. “Current status of application of foam concrete.” in Symposium - Concrete Research in India (CRI) on 14th September (2019), organized by IIT Madras, Chennai.
3. **Khwairakpam, S.,** and Gandhi, I. S. R. “Assessment of the potential of naturally available foaming agent for use in the production of foam concrete” 3rd International Conference on Innovative Technologies for Clean and Sustainable Development on 19-21 February 2020, organized by organized by National Institute of Technical Teachers Training & Research, Chandigarh and Maharaja Agrasen University, Baddi, Chandigarh, India.