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ABSTRACT FOR THESIS

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Experimental Investigations on Alkali Activated Foam Concrete with Impure Kaolin.

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Abstract

The main goal of this thesis was to produce foam concrete by using impure kaolinite coming from the mining industry of Finland. The purpose of making foam concrete was to use as light weight concrete in engineering applications such as thermal/ acoustical insulations and building blocks.

The experimental part consists of two different sets of experiments. The first set of experiments involve fresh properties to determine fresh density, viscosity, yield stress and aeration rate and to find the behavior of sample in the fresh state before curing. The second set of experiments was the harden properties including the compressive strength and water absorption. The purpose for testing was to find the effect of H_2O_2, molarity of NaOH and the effect of surfactants. The purpose of our study was to check the application of impure kaolinite in the production of light weight foam concrete. Furthermore, XRD and FTIR analysis were carried out to determine the crystalline structure and the chemical properties of the Alkali activated kaolinite. Polarized microscope was used to get insight about the pore structure and the effect of different parameters on the pores structure and size.

The main findings from the study is the possibility to use kaolinite without any processing and production of low temperature (400 ˚C) ceramic like concrete foam. The dosage of H_2O_2 was varied from 0.5 to 2%, it effected the hardened properties such as compressive strength as it goes from 20 MPa to 15 MPa. Furthermore, it effected the fresh properties of the material, especially the aeration rate. The aeration rate was increasing for all samples when the percentage of H_2O_2 was increased. The molarity of NaOH was an important factor that affected the water absorption. Microscopic studies showed that the pore structure varied between samples made without surfactants and those with surfactants, that explains the hardened properties of the aerated foam with impure kaolinite.

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PREFACE

This research was made for the University of Oulu in the Faculty of Technology and department of Fiber and Particle Technology. The main purpose of this study was to understand the performance of impure kaolinite without processing, as a source material for alkali activated foam. Our goal was to achieve good mechanical properties by modifying the pore structure with different surfactants like Triton and Cetrimonium bromide. The raw material was provided by different mines for the studies.

I am very thankful and appreciate help from my supervisor´s Priyadharshini Perumal and Päivö Kinnunen for offering me the chance to write this thesis and finding me a suitable topic. In addition to that, I want to thank my PhD colleague Katri Piekkari for her guidance and help, and for having all the patience. Furthermore, I want to thank everyone who helped me on the way, especially Marcin Selent and Tommi Kokkonen for their help with the XRD and Microscopic analysis, and the laboratory staff (Jarno, Elisa and Jani) for their help with everything. It has been an honor to be a member of the Fiber and Particle Engineering Research Unit.

I want to thank **Allah Almighty** the one and only one who created the universe and bestowed the mankind with knowledge and wisdom to search for its secrets, I set my sincere and humble thanksgiving before Him. Who favored and invigorated me with the fortitude and capability to complete my work? Allah the Almighty never spoils any effort. Every piece of work is rewarded according to the nature of devotion.

In the end, I want to thank my parents (Ishfaq Hussain and Nusrat Jabeen) for their endless support and their help in every aspects of my life and motivated me for my all tasks in the life.

Ali HASNAIN

SYMBOLS AND ABBREVIATIONS

°C	Degree in Celsius
AAM	Alkali activated material
Al	Aluminum
Al_2O_3	Aluminum oxide
BFS	Blast Furnace slag
Ca	Calcium
CaO	Lime (calcium oxide)
CO_2	Carbon dioxide
CS	Compressive strength
Fe_2O_3	Iron oxide
FTIR	Fourier transform infrared
FA	Fly Ash
g	Grams
h	Hours
K	Potassium
K_2O	Potassium oxide
KOH	Potassium hydroxide
LOI	Loss on ignition
M	Molar (mol/l)
Mg	Magnesium
MgO	Magnesium oxide

Na	Sodium
Na_2O	Sodium oxide
NaOH	Sodium hydroxide
Na_2SiO_3	Anhydrous sodium metasilicate
OH^-	Hydroxyl ion
OPC	Ordinary Portland cement
rpm	Rounds per minute
Si	Silicon
SiO_2	Silicon dioxide
S.D	Standard Deviation
Si/Al ratio	Molar ratio between Si and Al concentrations in a material
XRD	X-ray diffraction

1. Introduction

1.1. Motivation:

Geopolymers are one of the promising alternatives to replace the ordinary Portland cement and makes its place in the construction field. Geopolymers and cement are compared on different parameters such as on economy, strength, durability, energy demand and environmental-related effects. The environmental effects coming from geopolymers are far less then OPC with the use of lower energy, lower temperature, and less carbon emission. Different raw materials can be used to produce geopolymers such as Fly ash, Metakaolin, Kaolinite and we used kaolinite for our studies to produce foam concrete. In Finland, Mining industry is very big and the mining waste produced is increasing with time it was 123 million tons in 2016 given by statistics Finland in their report [1]. So, there is a need of reducing this amount of waste because it will help in the reduction of environmental effects and waste disposal management. Our motivation for this study is that, impure kaolinite application to produce the foam If this study will succeed then, there is a chance to avoid energy consuming treatment to convert the kaolinite to metakaolin for such applications in lightweight concrete production and ceramic making.

1.2. About this study:

The main purpose of this study was to use impure kaolinite to produce foam concrete to use as an alternative in construction industry. Our goal was to achieve the compressive strength comparable with OPC concrete, for applications on thermal insulation, water treatment and acoustic applications. The raw material was provided by a local kaolinite mine in Finland.

There were two sets of studies including fresh properties and harden properties. In the fresh properties, we studied fresh density, viscosity, yield stress, and aeration rate. The procedure for the study has been described in the fresh properties section. The comparison was made between three kinds of samples: without surfactant, CTAB, and Triton.

In the second set of hardened properties, we studied compressive strength, water absorption, XRD, FTIR, and microscopy. The procedure in detail given in the harden properties chapter and the same comparison between without surfactants, CTAB and Triton were made.

2. Foam Concrete:

2.1. Definition:

Foam concrete was invented in 1914 by Alysworth and Dyer. Foam concrete is a type of porous lightweight concrete made by cementitious binder mainly with the addition of fine aggregate [2], [3].

2.2. Method of production:

The preparation method of foam concrete consists of three parts.

1. Binder
2. Voids
3. Water
4. Surfactants

2.2.1. Binder:

The commonly used binder for the preparation of foam concrete is a cementitious binder that may or may not have the addition of the extra characters for special properties such as sand, lime and rapid hardening agents [4]. The industrial waste such as fly ash and BFS are used to replace the cement to make the process more sustainable with low environmental footprints and low emission of CO_2 [5], [6].

The different types of non-cementitious fillers are used to reduce the manufacturing cost of foam concrete. These fillers could be sawdust, diatomite, and bentonite which can provide better workability and the final strength to the concrete [7]. The highest percentage of these fillers should not increase the 50% of the paste otherwise there will be setting problems and segregation can take place [8].

2.2.2. Voids:

The introduction of pores or voids and the control of their size and distribution in the production of foam concrete is one of the critical parts which determine the foam concrete density and strength. The introduction of the voids can be done through several methods that can be listed down.

- By the addition of aerating agent which release gas and make pores in the aerated foam such as H_2O_2 mixed with cement paste.

- The other method is the addition of air bubbles (pre-formed foam) in a large number which can be added by using an organic foaming agent.

The properties of the foam concrete usually depend on the distribution of these pores and their sizes which mainly depend on the composition of mixture or paste and the curing process. These aerating agents happen to raise the paste or mortar [9]. The geopolymer without the addition of any aerating agent have more compressive strength as compared to the one having aerating agent. The compressive strength decreases with the addition of aerating agent because it makes the geopolymer as a porous material with less density. For example, A study was carried out by using different additives and analyzed the effect of additives on the strength of geopolymers. The compressive strength obtained by corn oil was 9.9 MPA with 26.6% of porosity and the strength with waste palm oil was 9.0 MPA with 21.3% and finally with starch it was 20.41 with 17.9%. The strength and porosity are inversely proportional to each other. [10]

2.2.3. Water:

Water is added to mix the foam concrete in a similar way as in the general concrete. The water which is used in concrete manufacturing should not contain any kind of oil or any other dissolved organic compounds otherwise it can affect the properties of fresh or harden concrete in a negative way [11]. The amount of water added in the paste formation is very critical and effective in the case of strength and mainly depends upon the composition of cementitious material available, filler and the requirement of workability [12].

The strength and density of the concrete depend on the amount of water and the number of induced pores in the concrete, so the low amount is favorable for the high strength. The low amount of water can be achieved by using some superplasticizer which helps in achieving the high strength of concrete [13]. It should be kept in consideration that there should be a balance between foaming agent, superplasticizer and the amount of water while making the concrete [14].

2.2.4. Surfactants:

Surfactant play an important role in creating an interaction between air and water. Surfactant help to form the stable foam. Surfactant can be two types non-ionic and ionic. Ionic surfactant is divided further in to two anionic and cationic. Foam stability also depend on the speed and amount of the adsorption of surfactant on air water interface [15], [16]. Zhang et al. try to evaluate the adsorption of surfactant by using non-ionic and both anionic and cationic and found that, there was no adsorption with non-ionic and there was partly adsorption with both and cationic and anionic surfactant [17]. In the recent past, petit et al. also made some experiment by using cement and cationic surfactant to determine the effect of surfactant but he did not find the remarkable effect of surfactant on it [18].

Normally, foam concrete or lightweight concrete is used for the insulation purpose of walls. Surfactants which are chemical substance are added to the mixture of precursors and water to control the pore size and shape. The strength of the foam depends on the pore size. If the pore size is big then the compressive strength of the foam concrete would be low and can be destabilized. The use of a surfactant can reduce the pore size up to 1.84 mm to 1.49 mm and can increase the compressive strength by 25% [19].

2.3. Properties of Foam Concrete:

The properties of Foam concrete are described below.

One of the most popular and important properties of foam concrete is thermal insulation which reduces the operational energy used over the life cycle of building [7]. The thermal conductivity depends on the factors such as porosity, the density of the material and the moisture contents in the paste for the saturation of pores [20].

The commonly discussed mechanical properties in the case of foam concrete are compressive strength and elasticity modulus. The factors which can affect the mechanical properties are the characteristics of fillers used, the fractions available in the raw material, water-cement ratio (w/c) and the method of curing the foam concrete. The number of pores and their sizes also play a part in affecting the mechanical properties.

Shrinkage takes place at a high degree in the foam concrete as compared to the normal OPC concrete which is 5-10 times and the reason behind this is the high ratio of water in it. The estimation of subtraction takes place after one year. It can fall below the range of 0.1-0.36% [21].

3. Alkali Activation:

Alkali activation has a very old history, first presented by German engineer Kuhl in 1908, by giving the concept of the reaction between alkali and aluminosilicate precursor to form solid material which had the same kind of structure like ordinary Portland cement (OPC) [22]. Further studies were carried out by Purdon in 1940 tested 30 samples by using blast furnace slag (BFS) as a solid precursor in the alkali system of NaOH and $Ca(OH)_2$ and achieved the strength very close to Portland cement [23], [24]. The amount of water added, and the selection of proper alkali activation agent should be specific to get the required properties. Alkali activated materials (AAM) came into existence because of the shortage of cement in the Soviet Union and China, as an alternative to overcome the problem by using readily available material such as metallurgical slag. After Purdon, there was a break until the 1970's when Davidovits started working in France and gave alkali-activated aluminosilicate material the name geopolymers [25], [26].

Alkali activated material is a very broad term including different activation agents and solid precursors' Activation agents can be alkali hydroxide, silicates, carbonates or sulfates, etc. The solid precursor can be calcium silicate or aluminosilicate depending on the requirements. The reaction takes place between solid precursor and alkali in the presence of alkali activation agent to form geopolymers. The most commonly used precursors are BFS, fly ash (FA) and metakaolin, etc. [27].

Geopolymerization which is exothermic reaction is the chemical integration of minerals by covalent bonding which mostly involves aluminosilicates [28]. Any raw material having silica and alumina can act as a geopolymer precursor and should be soluble in the alkaline solution to form a geopolymer [29]. The alkali components which act as activation agents are present in the first group of the periodic table and it is also called aluminosilicate binders [30].

Geopolymerization have very interesting industrial applications (Thermal insulation, lightweight material, Acoustic application) in addition to reduce industrial waste and convert them in a useful and sustainable product [28]. Based on the chemical composition the AAM can be divided into two groups.

- The precursors which have a high amount of calcium such as Blast furnace slag or Fly ash produce calcium silicate hydrate gel when reacted with alkali activation agent solution. In the strict sense of the geopolymer definition, these fall outside of the category, but are alkali activated materials.

- The other group of geopolymers has low contents of calcium e.g. metakaolin when these materials reacted with alkali it produces a porous structure that has high thermal resistance and can be used for acoustic applications [31].

The AAM or geopolymers specifically aerated are different from OPC as they have different reaction chemistry and AAM have high alkali contents in it and they are light in weight as compared to Portland cement with high thermal resistance [32].

3.1. Aluminosilicate Precursors:

In the case of geopolymers, the commonly used raw material is the aluminosilicate material. Different materials containing aluminosilicates coming as industrial waste from different industries can be used as a raw material. These sources do not have the same quality or standard and they are not available abundantly to produce geopolymers. The most commonly used raw materials containing a high amount of aluminosilicate for geopolymers are clay and fly ash. If we compare fly ash and clay, fly ash is better in the case of workability and requires less amount of alkali activator agent [33]. On the other side, fly ash coming from different sources may have entirely different setting behavior [34]. Kaolinite is usually added if there is a need for gel formation of desired properties because it enhances the dissolution of Al [35].

3.2. Mechanism of Geopolymerization:

Geopolymerization produces a three-dimensional macromolecular structure. The main chemical reaction takes place between aluminosilicate oxides and alkali to provide a bond Si-O-Al. This achieved by the calcination of alumino-silicate hydroxide (Si_2O_5, $Al_2(OH)_4$) following the reaction.

$2(Si_2O_5, Al2\ (OH)4) \rightarrow 2(Si_2O5, Al_2O_2)\ n + 4H2O$

Or

The condensation of SiO and Al_2O vapor according to the given reaction [36].

$4SiO\ (vapour) + 2Al_2O\ (vapour) + 4O_2 \rightarrow (Si_2O_5, Al_2O_2)\ n$

The reaction strategy of geopolymerization of minerals by condensation given by Xu et al. [35] is shown below.

Al–Si material (s) + MOH (aq)+Na_2SiO_3 (s or a) (1)

Al–Si material (s) + [$M_2\ (AlO_2)\ x\ (SiO_2)\ y.\ nMOH.\ mH_2O$] gel (2)

Al–Si material (s) + [$Ma\ ((AlO_2)\ a\ (SiO_2)\ b)$]. $nMOH.\ mH_2O$ (3)

In the reactions (1) and (2), the amount of Al-Si material is used depends on the size of the particles, the degree of dissolution of Al-Si materials and the concentration of the alkaline solution. Gelation [Mz (AlO_2) x (SiO_2) y $\leq$ nMOH mH_2O] is based essentially on the degree of dissolution of the aluminosilicate materials, while geopolymers of an amorphous structure are formed during the reaction (3).

3.3. Why Alkali Activation?

Cement and concrete are the basic building materials all over the world and is one of the economic determination factors nowadays for a country. It accounts for the US $ 3.3 trillion to world economics given in 2008 [37]. The largest production of these two materials resulted in a huge environmental impact due to the production of hazardous material and flue gases during the process. The cement production generates 5 to 8% of world CO_2 emissions which is very alarming and dangerous for the environment [38], [39]. There is a rapid increase in the demand for cement day by day mostly in developed countries and due to this, there is a need to find alternatives for these building materials and to decrease the level of CO_2 in the earth, atmosphere.

The best practice available is the use of alternative fuel for the manufacturing of cement, use of cement at optimum level in the concrete and recycling will decrease the cost as well as the emission of CO_2 which eventually will decrease the environmental impacts [40]. One of the benefits of using AAM over the cement is the reduction of CO_2 to a large extent. AM is one of the best alternatives for construction materials given environment impact as well as AAM is very economical due to the need for low temperature and low usage of energy to produce AAM as compared to OPC, which makes it economical [41]. Alkali activation also converts the waste material from industries to a useful product that otherwise would be landfilled. For instance, the use of metakaolin, FA or clays [42].

AAM are struggling to make their way in the market on commercial level because the environmental impacts cannot drive the use of AAM in the construction field, even though the whole world is now focusing on the issue of global warming and the use of green products to decrease the environmental impacts. But there is a need to make the AAM production economical as compared to cement in addition to it [39]. The main issue in achieving this is the availability of raw material which should be available locally and abundantly so it would be less expensive as compared to cement will make it economic and competitive.

The other concern of the cement or construction material dealers is the strength and durability of AAM which should be comparable to cement [43].

The nanostructure of the AAM mainly depends on the type of precursors. The highly calcium contained system has calcium silicate hydrate like structure while the system with low calcium such as metakaolin has a disordered 3D structure [44]–[47]. The most commonly used techniques to study the nanostructure of AAM are FTIR and NMR. These studies are carried out to understand deeply the structure of AAM.

Further, the microstructural analysis of AAM is carried out by scanning electron microscope (SEM) and transmission electron microscope (TEM). These techniques are used to identify the number of fractions available in the precursors and the number of unreacted precursors. They are also able to analyze different phases available in the given precursor. These techniques give quantitative analysis and give the idea to improve the microstructure of the product [48]–[50].

The other popular technique to study both nanostructure and microstructure is tomography which gives three-dimensional insights to the AAM structure and allows viewing the pore structure of AAM [51]–[53].

3.4. Advantages of using AAM over the OPC:

Alkali activated material is invented as the alternative for the Portland cement and concrete to reduce the environmental footprints. Cement is the number one product in the world, which is the basic building material for construction, is one of the biggest sources of CO_2 emission. Concrete is the second most-produced product which mostly manufactured by using Portland cement [22]. AAM or geopolymers as compared to cement have a low manufacturing temperature (700 °C) needed to produce ordinary Portland cement (1450 °C). Cement has its monopoly in the market because it has very long durability, strength and uniform structure with mechanical, thermal and chemical resistance [54].

There are different types of raw materials available to produce AAM or geopolymers, depending on the contents available in the raw material such as calcium aluminosilicate, sulfoaluminates, and magnesium-based precursor, etc. The main reaction takes place between binders such as fly ash, blast furnace slag or metakaolin and the alkaline solution such as NaOH sodium silicate to produce strong and insoluble AAM with less amount of CO2 [55].

The BFS and Fly ash can achieve comparable performance as that of cement and concrete with low environmental impacts and fewer emissions, sometimes for special applications such as resistance to sulfates [42]. Fly ash and BFS may be used as a replacement of cement and concrete or could be blended with Portland cement. In both conditions, the requirement of an alkaline activator would be different. If there is a large amount of cement and less amount of fly ash or BFS then we can use cement standards for production. In other conditions, if there is a large amount of FA or BFS such as 95% binder and 5% cement then we would need different standards for an alkaline solution with a high pH value [56].

In 1950 Glukhovsky produced AAM by using low calcium aluminosilicate precursors as the alternative for cement for the Soviet Union using alkali activation agent. Later in the 1980s, In Europe, the foam concrete was produced and named as F-concrete on a commercial scale [50], [51].

Further research was carried out and the high strength concrete produced in the 1990s and given the name of Pyrament in North America. If AAM is used in the production of concrete, it can result in almost the same technical properties as compared to the cement. This can be applied in the construction field to get properties such as thermal resistance, lightweight and durability [55].

3.5. Synthesis of alkali activated foam concrete:

Two commonly used foaming methods are mechanical and chemical foaming to introduce pores into foam concrete. There is a minor difference in both methods, pre-foaming is added during the mechanical method and chemicals are added in the chemical method. Surfactants and chemicals control the pore structure and size and keep them confined in the foam concrete body. The foaming decreases the overall density of concrete. The pore size and shape are more stable and equally distributed in mechanical foaming as compared to chemical foaming in which the pore size is big and pore shape is non-uniform [57]. The overall foaming process can be seen in the following fig.1.

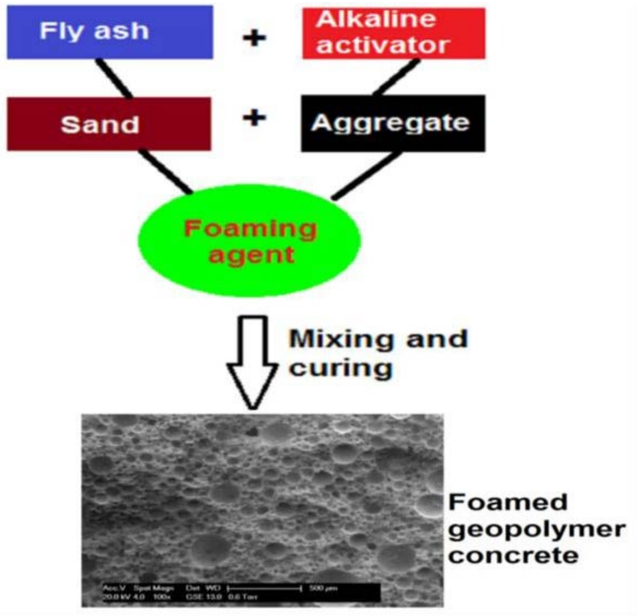


Fig. 1 Synthesis of foamed geopolymer concrete [57]

3.5.1. Differences between solid and foamed geopolymers

If we consider the chemistry of geopolymers, the phase formation after the introduction of pre-foaming agent or the gas-releasing agent is unknown. For example, considering Al powder, which is one of the commonly used gas-releasing agents, used in both OPC foam concrete and geopolymer foam concrete [58], [59]. The amount of AL powder added, and its reactivity will have a great impact on the chemistry of geopolymers at the micro and Nano structural level because it releases aluminates and H_2 gas and the time available for aluminates to integrate also plays important role in achieving good geopolymer structure [60].

3.5.2. State of the Art (Literature):

Several studies have been carried out to understand the chemistry and behavior of the foam concrete or aerated concrete. Table 1 gives detailed information of how aerated concrete behave with different condition of raw material, alkali activation agent and aerating agent to achieve different strength and density. The curing temperature ranges from room temperature to 85 ° C. The most commonly used raw material was Metakaolin and NaOH was the common aerating agent except in some cases where they used Na_2SiO_3 in addition. the strength ranges from 1.1 to 5.5 MPA.

Table 1: Review on the behavior of Metakaolin in different conditions

Author	Material	Paste/ Mortar	Surfactant	Aerating Agent, (dosage%)	L/S ratio	Na-Si/ NaOH	Method of curing	Strength (MPa)	Density kg/m^3
Palmero et al. [61]	MK$^\#$	Paste	No details	H_2O_2 (1-6)	0.57-0.6	1.64	RT* or 65°C for 24 h until testing	2 - 5.2	330-690
Samson et al. [62]	MK + FA	Paste	commercial surfactant	H_2O_2(0-2)	0.49-0.84	3.83-7.10	20°C for 24hr	53.2-53.7	259-507
Dembovska [63]	Metakaolin glass waste	Paste	No details	Al $(NO_3)_3$	0.63-0.75	0.11	24h at 80°C	1.1- 2.0	380 - 470
Wu et al. [64]	MK + FA	Paste	Calcium stearate (CS)	H_2O_2(4)	0.2-0.3	1.2	25-85°C for 12 hr	1.3-1.8	249-257
Bai et al. [65]	MK	Paste	No details	H_2O_2 (3)	-	-	75°C for 24 hr	2-4	-
Zhu et al. [66]	Mk	Paste	No details	Al+IBA$^\times$ (0.03-0.07+7.5-17.5)	0.22	3.89	RT for 3 days	5.5	860

IBA$^\times$ (incineration bottom ash) Mk$^\#$ (Metakaolin) RT*(Room temperature)

4. Mine Tailing

The material used in the study is not mine tailing, but since it is close in composition to mine tailings, it is worth making the connection here. Mine tailings are usually in the form of powdered form in the range of (1-600 μm) particle size, which are obtained after getting valued minerals removed from mother ore and the processed water. For example, through copper mining, we can obtain 95-99% tailing in the form of crushed and ground powder. Some 10 or 20 years ago, because of the degrading of ore grades, the more gridding was required for the complex ores that are producing more tailings per unit of ore obtained. This requires careful investigation by the society and regulators. [33], [67]

4.1. Disposal issues for tailings

The properties of tailings depend on the type of ore they are extracted from. Sometimes there are some metals and acids, which are going with waste, are needed to be neutralized before disposal. The solid waste from tailing can also have sulphide which has environmental impacts. The deposition of sulfide mineral, especially in case of fool's Gold (Iron pyrite) can react with incoming rain or snow, melts to make sulphuric acid and this can discharge metals from tailing. In solid tailings, there is a probability of the presence of fine particles and sludge which can affect the strength of tailing. It may also contain chemicals, acids, leaching agents, which also take part in environmental hazards**.** Dam failure is one of the significant incidents related to tailing disposal [34].

There are several methods of waste disposal including dams that cross valleys or hills, high-altitude slopes, stacks of dry-thickened dumps, ground filling in open-pit mines or unused underground mines area, and direct disposal in forest areas, rivers, lakes, and oceans (surface and submarines). Backfill or pit storage in unused sections of the mine will be used in multiple locations, but mine operations cannot be restored. The disposal directly into the ocean is one of the popular methods of disposal for many years in many regions of the world. For example, Batu Hijau Copper-Gold Mine on Sumbawa Island, Indonesia, operates an Offshore Tailings Camp (TSSP) to remove tailings in Senunu Canyon (offshore) [58]. There are many methods available for the disposal of tailing based on sustainable development with a focus on the reduction of waste and improvement in it. The present system of tailing management includes the water quality control of discharge water, dust management and the available landfills within the area of mine [59]. The environmental impacts of waste coming from tailing can be reduced by modifying the mining plant design rather than operation [60]**.** Mostly, the environmental impacts depend on the type of ore of mine and chemical and physical operations during its production. A lot of research has focused on reducing the negative environmental impacts of mining and tailing waste.

4.2. Mined clays as raw material:

Mine tailings are a difficult raw material for industrial exploit due to their highly varying material properties. Due to the complicated mineralogy of the mining site, the refining process and sedimentation in the tailings pond, mine tailings are highly heterogeneous concerning chemical composition, mineralogy and particle size distribution. However, most industrial processes require homogeneous raw materials to maintain a standard level of quality for their products [68]. Therefore, mine tailings, such as other waste materials, require a higher amount of characterization than fresh raw materials [69].

Geopolymerization is a relatively robust process, and quite insensitive to the variation of non-crucial components. Therefore, mine tailings with a suitable Si/Al-ratio are potential aluminosilicate sources for geopolymerization. For example, tungsten mining side streams, oil sand tailings, and copper mine tailings have been studied as potential aluminosilicate precursors [68]. The reactivity of the tailings depends mainly on chemical composition and mineralogy, and it is typical for tailings to have too high Si/Al ratios for a successful alkali activation [70]. However, some of these materials can be activated better by the addition of a more reactive mineral, such as kaolinite [29]. The most ideal minerals for geopolymerization are 1:1-layer lattice aluminosilicates. Tailings with non-reactive mineralogy can often be pretreated to increase their reactivity [68]. All in all, due to the complex nature of mine tailings, the suitability of a given mine tailing cannot be defined with certainty theoretically, but empirical testing should always be done [29].

5. Materials and Methods:

5.1 Materials:

5.1.1 Kaolinite:

The main raw material used for the study was impure kaolinite obtained from Finnish mining industry. The raw material was dried at 100 ˚C. Dried impure kaolinite was milled with one part of steel balls and two parts of materials in the container of 10 Kg for 10 mins using ball mill, to get the powder form of the material.

The particle size distribution of the milled kaolinite is given in the table 2. The size of the particle’s ranges from 2.695 µm to 299 µm. It also gives the percentage of different size particles in the raw material. The instrument used to measure the PSD was Beckman Coulter LS 13 320. The graph was made between particle diameter, differential volume and cumulative volume as shown in the figure 2.

Table 2: Particle size distribution for impure kaolinite

Particle size(µm)	2.695	7.816	21.86	102.8	130.9	213.1
Percentage Existance	<10%	<25%	<50%	<75%	<80%	<90%

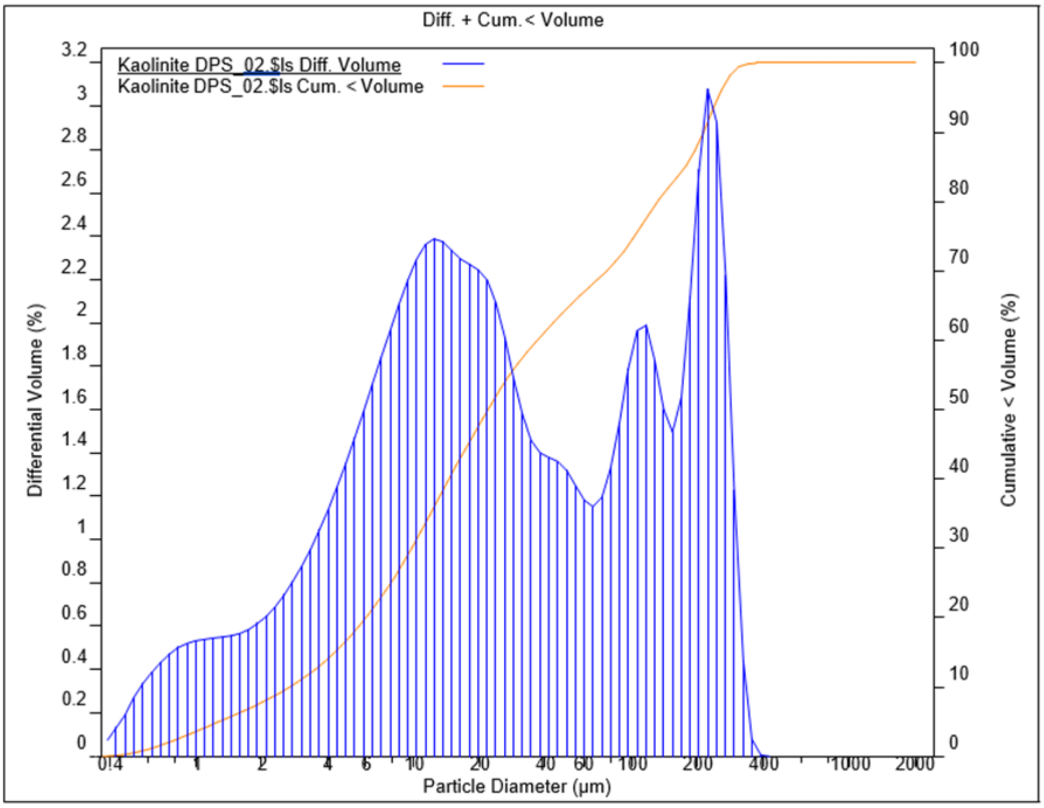


Fig 2. Particle size distribution graph with respect to differential and Cumulative volume.

5.1.2 Alkali activator

An alkali activation solution was made by using pellets of NaOH supplied by VWR chemicals for three different molarities viz., 5M, 10M and 15M. The pellets were mixed in distilled water by using electric mixer and it was kept for 3 hours to cool down to room temperature.

5.1.3 Surfactants

Cetyl trinethyl ammonium bromide (CTAB) and Triton from VWR distributor chemical were used as surfactants for this study. The fig 3 shows the pellets of NaOH, final powder form of kaolinite, CTAB and Triton, which were used in this study.

5.2 Methodology

The methodology carried out to make samples has two sets of experiments for Fresh properties and Harden properties. There was a minor difference in the weight of samples for two sets. For fresh properties samples,50 g of kaolinite was used to make the samples with 0.55 and 0.65 water – binder (W/B) ratio with 5M and 15M of NaOH solution and 0.5% of CTAB and Triton surfactant. For harden properties,100 g of kaolinite was used with 0.55 W/B ratio with 5M,10M and 15M of NaOH.

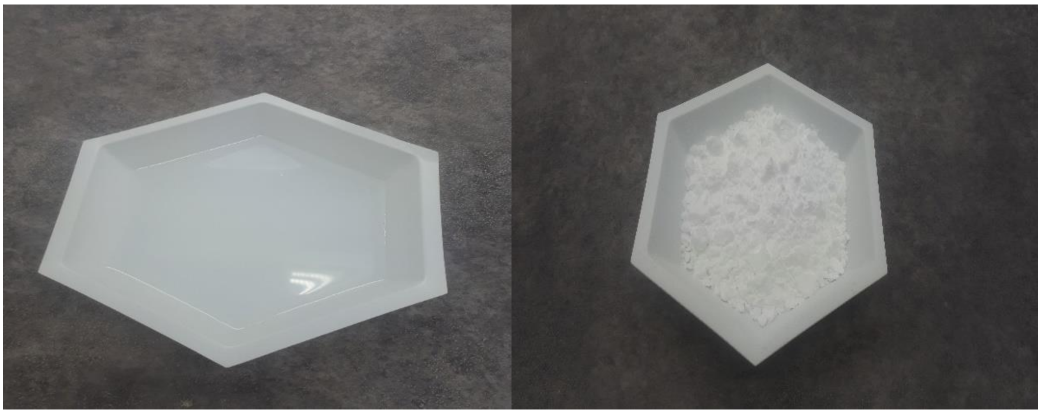
Table 3: Oxide composition of Kaolinite.

Oxides (%)	SiO_2	Al_2O_3	MgO	CaO	Fe_2O_3	K_2O	Na_2O	other	LOI
Impure Kaolinite	72.52	16.43	0.824	0.047	1.896	3.053	0.078	0.742	4.417

The process of the sample production was that, kaolinite was mixed with fixed water content as designed in w/b ratio for 2 mins, followed by the addition of H_2O_2 and surfactant and mixed for another 2 mins by using the shear mixer at the speed of 3000 rpm. For harden properties, similar mixing procedure was followed and after mixing, the fresh paste was filled in 5 cm cubic specimens as shown in the figure 4. They were kept for 2 hours at room temperature for the aeration to complete and 22 hours under the temperature of 60 °C. After that, 28 days for curing were carried out before testing them for harden properties. Furthermore, the samples were heated under 400°C for 1 hour in furnace to produce the low temperature ceramics and the hardened properties were evaluated.

a) Kaolinite

b) NaOH pallets



c) Triton d) CTAB

Fig 3. Raw materials used for the studies

Fig 4 .5×5×5 molds used to make the samples.

5.2.1. Fresh Density and Aeration Rate:

To measure the fresh properties including the fresh density and aeration rate, glass beaker with a scale it was used. The arrangement can be seen in the figure 5. Fresh properties were measured before hardening of the paste sample within 30 minutes of mixing them. There were 48 samples studied for the fresh properties. The samples are compared for the effect of H_2O_2, molarity of NaOH and W/B ratio. The raw material was used in homogenous finely ground powder form. The sample production process is already explained in method and material section in detail. The fresh density was measured using the measuring balance shown in the figure 5. The aeration rate was measured by reading the scale on the beaker.

Fig 5. Arrangements to measure the fresh properties (Fresh density and Aeration rate).

5.2.2. Rheology:

Mostly the rheological studies carried out to determine the deformation under certain force exerted on a specific material. The capacity of the deformation of material usually depends on the properties of a material. For example, light material such as air can be flown by applying very small force as compared to some heavy material such as honey [71]. For our studies, the raw material was dried for 24 hours at 100 ˚C and ground for 10 mins at the speed of 1400 rpm in vibrating disc mill. Then we made 48 samples of 16 mixes containing a reference, CTAB, and Triton. The dry powder was mixed with the alkali solution of NaOH for 2 mins at 3000 rpm using the shear mixer. After 2 mins H_2O_2 and surfactant were added, and shearing mixing carried for 2 more mins to get the smooth paste. The amount of raw material powder was 50 g. The molarity used was 5M and 15M of NaOH with 0.55 and 0.65 of the water binder ratios. The range of H_2O_2 was from 0 to 2%. The geometry used for the study was vane concentric cylinders, Peltier steel with shear viscosity ramp up and ramp down. In the conditioning step, the temperature was kept at 22 º C with a soak time of 10 secs. The pre-shear applied at the rate of 100 1/S for the duration of 30 secs. After the preconditioning, both the ramp cycle was carried out for 180 s each. To compare the viscosity of the samples a single point in share rate was chosen. The chosen point of shear rate was around 24.4 1/s. Herschel-Bulkley model was used to analyse the data.

5.3. Hardened Properties:

In the harden properties, the following properties were tested for analyzing the behavior after the curing of the samples.

- Compressive strength
- Water absorption
- Density

5.3.1. Compressive strength:

Compressive strength is used as an important parameter to determine the material strength especially concrete. It can be defined as the maximum force a material can bear without deformation or fracture. The compressive strength can be calculated from,

$$CS = F / A$$

Where, F shows the force which applied gradually on the cross-sectional area of the sample. Sample can be in different shapes such as cubical, cylindrical and spherical. On the application of the force, some of the material can fracture but other will only deform. Compressive strength is a good parameter usually used in the designing field. It is mostly used in the construction field for building designed by Engineers.

Two types of fracture can be followed by material either ductile failure or brittle failure and it depends on the material nature. The compressive strength can be affected by the measurement procedure and environment of the test. Compressive strength always described as a comparison of some standards.

Concrete and ceramic material have the highest compressive strength than other materials which can have more tensile strength. Compressive strength is the main parameter to determine the quality of concrete material. The range of the compressive strength can be in between 17.23689 MPa 27.57903 MPa depend on the requirements [72]. To measure the compressive strength, we used the machine zwick/roell z010 for our samples shown in the figure 6. The maximum force limit for the compressive strength machine was 10 kN. Our samples were in the dimensions of 5 × 5 × 5.

Fig 6. Zwick/roell z010

5.3.2. Water Absorption:

For water absorption we tested 33 samples to analyze the change of percentage absorption by changing the molarity and H_2O_2 contents in the sample. Dry weight of the specimens was measured after exposing them at 100 ˚C for 24 h. The specimens were weighted for wet weight after immersing them in water for 24 hours. water absorption of the mixes was compared based on their H_2O_2 percentage and molarity of NaOH. The experimental setting for water absorption is shown in the figure 7, given below.

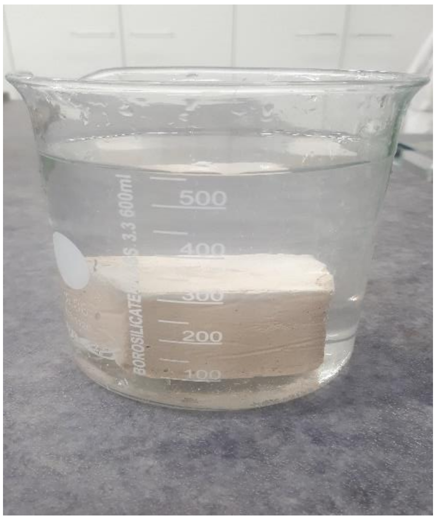


Fig 7. Water absorption settings.

5.4. XRD:

X-ray diffraction is a very useful non-destructive tool to determine the characteristic of material in the wide range including fluids, powder and crystalline materials. XRD have wide range of applications in the industry and research field to characterize the material. It determines the phase of material and it also give the quantitative data of the material. It is very useful to determine the three-dimensional atomic structure of the material. The principle of the XRD is based on the bragg equation as given below,

$$n\lambda = 2d\sin\theta$$

In this equation, n is the number, λ determine the wavelength of the x-ray beam which is used to bombardment of sample for the XRD studies. θ is the angle of diffraction of the x-ray beam and d is the spacing between layers of the material. [76]

5.5. FTIR:

FTIR is used to determine the properties of chemicals including organic and inorganic. It can also give the quantitative analysis of unknown material which can be solids, liquids or gases. FTIR term came into existence because of the way of collecting data from it by converting infrared to spectrum. It is usually used to determine the chemical bonding of the atoms in the material. The infrared adsorption spectrum produces and the wavelength of light which absorb determine the characteristics of chemical bond between atoms. [77]

Principle of Operation of the Fourier Transform Infrared Spectrometer Infrared spectra (IR) involve the investigation of the interactions between matter and electromagnetic fields in the IR region. In this spectral range, electromagnetic waves couple with molecular vibrations. Molecules are excited by absorption of infrared radiation to a higher vibrational state. Once absorbed, the infrared frequency would interact with the molecule at some frequency. IR spectroscopy is therefore a very powerful technique that provides fingerprint information on the chemical composition of the sample. By means of IR spectroscopy, qualitative and quantitative analyzes of fiber samples can be carried out. FTIR spectrometry is the type of most analysis techniques available in the laboratory. FTIR works on the principle of Fourier transformation. [78]

6. Results and Discussion:

6.1. Fresh Properties of Foam concrete:

Fresh properties are the properties that were measured before hardening of the concrete.

The following properties were studied in this work:

- Fresh Density
- Aeration rate
- Viscosity
- Yield stress

There were two sets of fresh properties, set 1 includes fresh density and aeration rate of the sample and Set 2 consist of viscosity and yield stress, the rheological properties of the paste.

6.1.1. Effect of H_2O_2 dosage on Fresh Density and Aeration rate:

To measure fresh density and aeration rate, 50 g of the sample was used with 0.5% of surfactant , and measurement was taken after regular intervals: 5, 10, 15 and 20 mins for fresh density and 2, 4, 6, 8, 10, 15, 20 mins for aeration rate. There were 16 mixes and 48 samples for which fresh density and aeration rate were measured, and the results for CTAB, Triton and the reference were compared. In fig 8: the effect of H_2O_2 dosage is presented and it shows that the fresh density of reference material and CTAB is decreasing linearly with increasing percentage of hydrogen peroxide in the sample, while the fresh density with triton was decreasing dramatically from 0 to 1 % of H_2O_2 but after this it became constant until 1.5 %. Furthermore, the effect of hydrogen peroxide on the aeration rate has been shown in Fig 8: in which it can be seen clearly that the rate increased by increasing dosage for all the cases. The increment was a bit higher in the case of reference, but the behavior was quite similar in the case of CTAB and Triton. With 0.5 % hydrogen peroxide the behavior was the same for all the materials, but it starts changing from 0.5 % until 1.5 %.

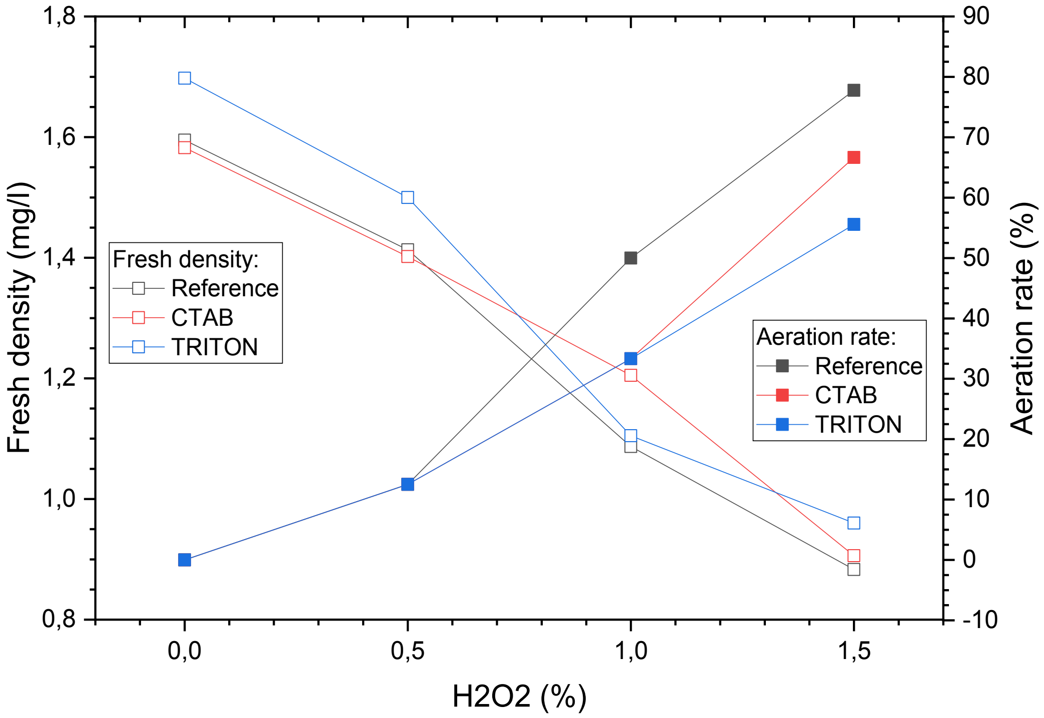


Fig 8. Effect of H_2O_2 dosage on fresh density. (W/B:0.55, NaOH:5)

6.1.2. Effect of Molarity on Fresh density and Aeration rate:

In figure 9, the effect of molarity on fresh density and aeration rate is shown. It is noticeable that the fresh density of reference and Triton does not change with the change of molarity, but there is a notable change for CTAB which decreases with the increase of molarity. The effect of molarity of NaOH is similar with the use of both the surfactants, CTAB and Triton on aeration rate and both are decreasing by decreasing molarity from 15 to 5. This might have happened because the aeration rate slowed down by surfactants for their role in structural arrangement and pore distribution. In the case of reference, the effect is the opposite, and it is increasing by decreasing molarity, which happened because the aeration is uncontrolled plus the viscosity of the solution is decreasing by decreasing molarity. As a result, the aeration rate became high.

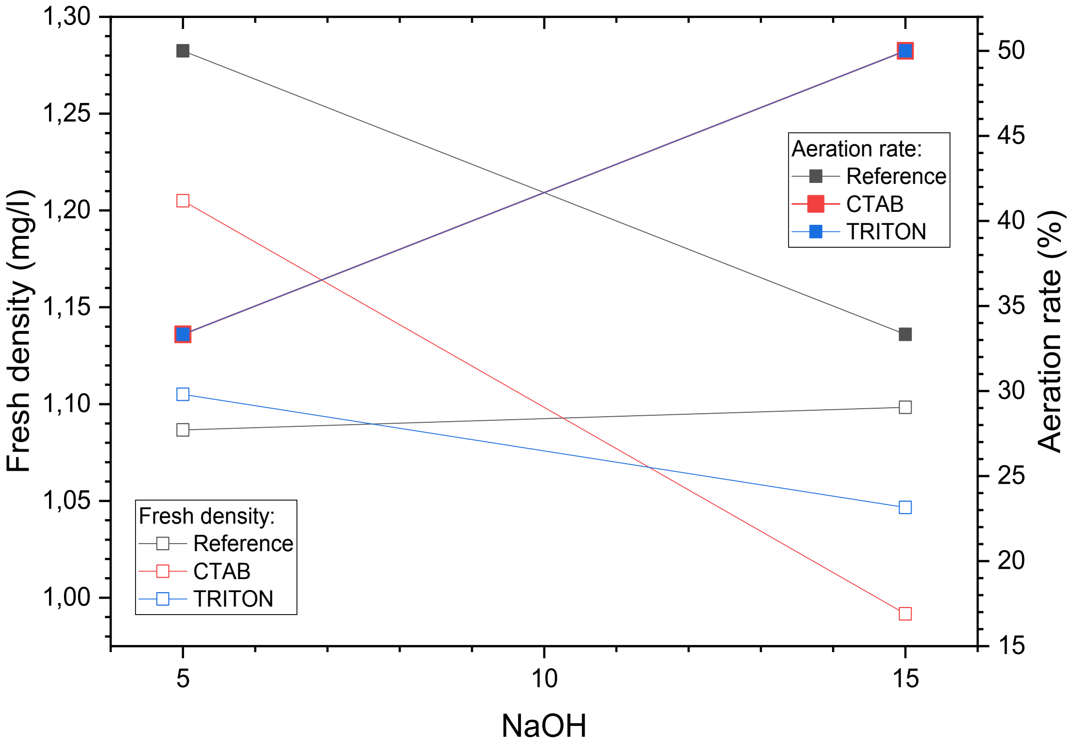


Fig 9. Effect of Molarity of NaOH on Fresh density. (W/B:0.55, H_2O_2:1%)

6.1.3. Effect of Water binder ratio on Fresh density and Aeration rate:

In Figure 10: the effect of water binder ratio on fresh density and the aeration rate is displayed, and it can be seen clearly that the fresh density is increasing with the increasing water binder ratio in each case. However, the fresh density for reference and Triton is almost same with 0.55 and there is the larger difference with 0.65 water binder ratio. It is clearly shown that the aeration rate is decreasing with increasing the w/b ratio. The reference and CTAB have a constant decrease with the increment of the w/b ratio while the decrease in aeration rate for triton is comparatively fast and there is a large difference in starting and ending point as compared to others two.

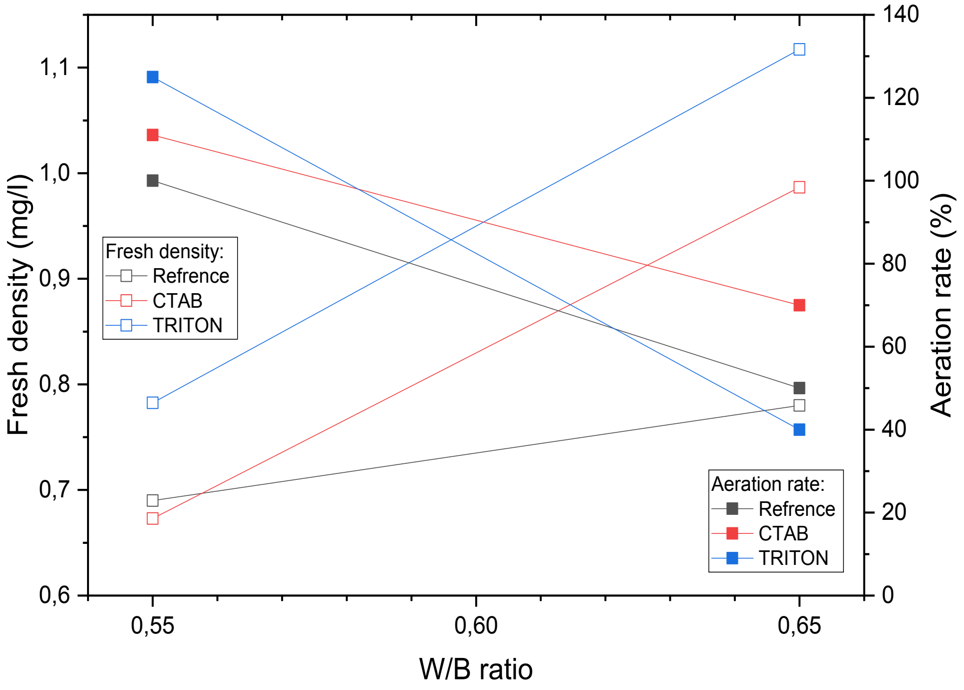


Fig 10. Effect of water binder ratio on fresh density. (NaOH:15, H_2O_2:2%)

6.1.4. Effect of H_2O_2 on viscosity:

In figure 11: the effect of H_2O_2 on viscosity is presented measured by rheometer on specific shear rate point 24.4 1/S as a reference point and the result was compared for without surfactant, CTAB, and Triton. There is a linear decrease in the viscosity of CTAB and Triton from 0% to 2%. The behaviour of the reference is a bit irregular as the viscosity was constant from 0 to 0.5% and there was a downfall until 1% of H_2O_2. There is a surprising increment in the viscosity from 1% to 2%. The decrease in the viscosity with an increment of H_2O_2 dosage is happening because of the increase in the number of pores which make it light and decrease the density.

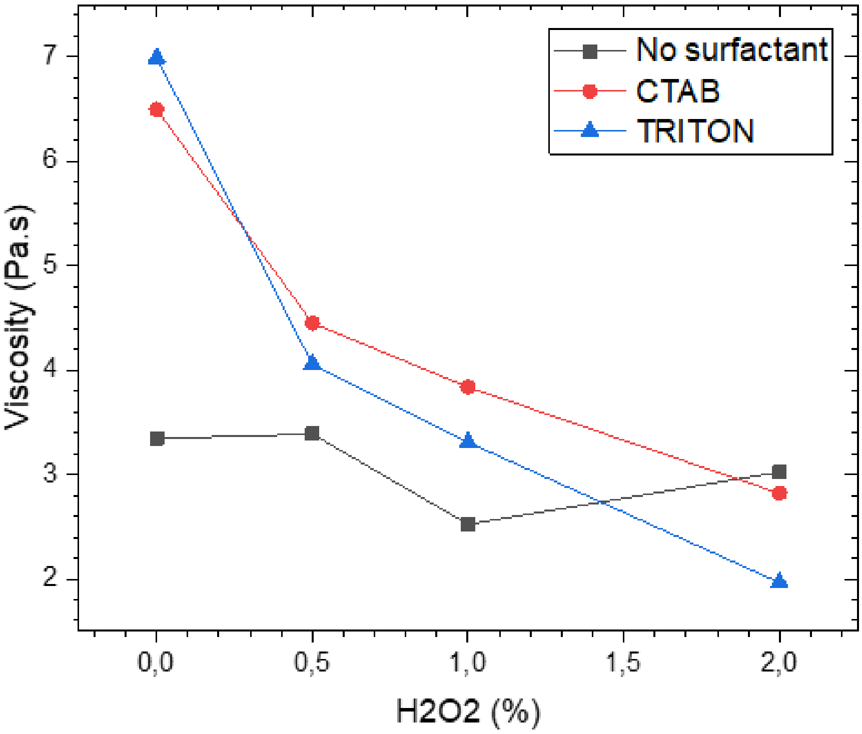


Fig 11. Effect of H_2O_2 on viscosity. (NaOH:15, W/B:0.65)

6.1.5. Effect of Molarity on viscosity:

Figure 12: shows the effect of molarity on the viscosity for the samples with reference and with surfactants CTAB and Triton. There is a decline in the viscosity of the CTAB and reference with the increment of molarity. There is a small difference between the starting and ending value of viscosity for CTAB but there is a large difference for the reference between endpoints. In this case, the behaviour of triton is quite different with a very small decrease in the viscosity with the increase of molarity value.

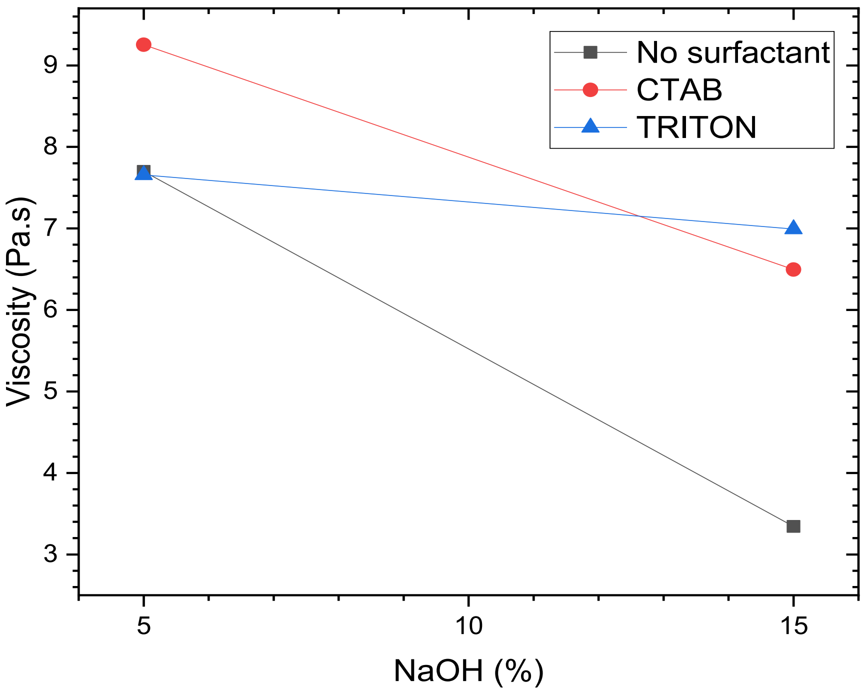


Fig 12. Effect of Molarity of NaOH on viscosity. (W/B:0.65, H_2O_2:0%)

6.1.6. Effect of W/B ratio on viscosity:

The figure 13: illustrates the effect of the W/B ratio on the viscosity of the material without surfactant (reference), CTAB and Triton. The behaviour for all of them is almost similar and there is a constant reduction in the value of viscosity with the rise in the value of the W/B ratio. The reason behind this is that the samples are becoming thinner and less viscous with the introduction of a large amount of water.

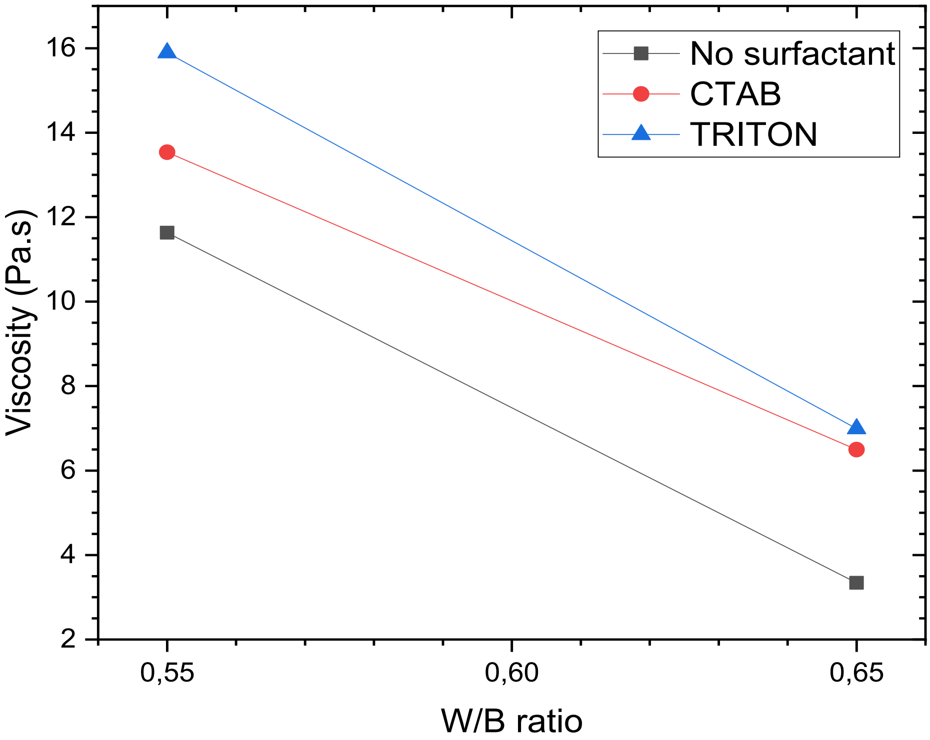


Fig 13. Effect of W/B ratio on viscosity. (NaOH:15, H_2O_2:0%)

6.1.7. Effect of the H_2O_2 on the yield stress:

The figure 14: shows the effect of H_2O_2 on the yield stress of the samples. The decreasing trend can be seen in the CTAB while the yield stress increased for the reference and triton. The maximum difference in initial and final value can be seen in the Triton. The behaviour is very different for each sample so the exact reason for this is not understandable and needs further investigations. The yield stress decreased for Triton decrease from 0-0.5 % of hydrogen peroxide but after that it increased surprisingly until 2% of H_2O_2.The yield stress decreased for the CTAB up to the 1% of H_2O_2 and then there is a stable increase in the value of yield stress.

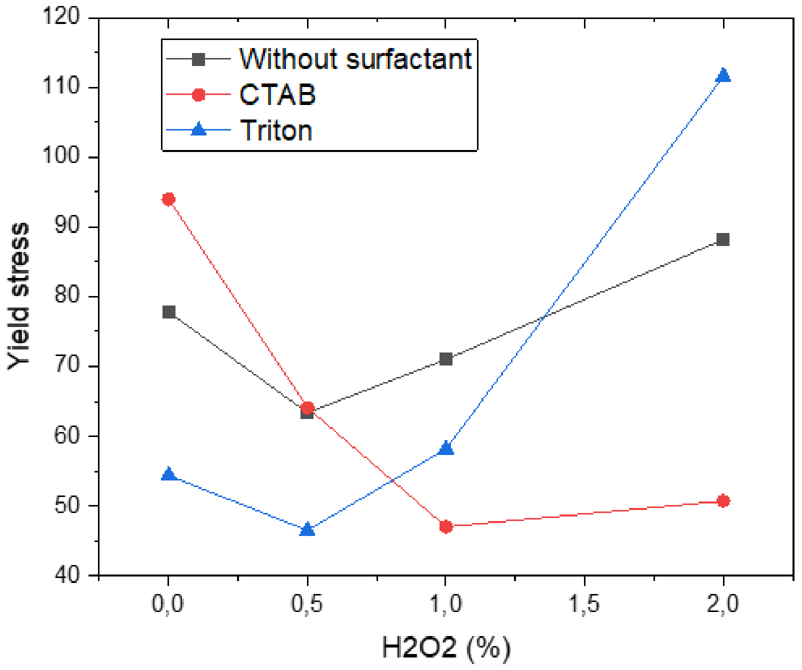


Fig 14. Effect of the H_2O_2 on the yield stress (NaOH:5,W/B:0.65)

6.1.8. Effect of W/B ratio on yield stress:

The effect of w/b ratio on the yield stress can be seen in the following figure 15: and it is shown that the yield stress decreases with increasing the water binder ratio for all three kind of samples. The major decrease in the value experienced by CTAB and Triton. The behaviour of the reference is quite different from the others two and the value of yield stress is same for both water binder ratio in this case with slight decrease.

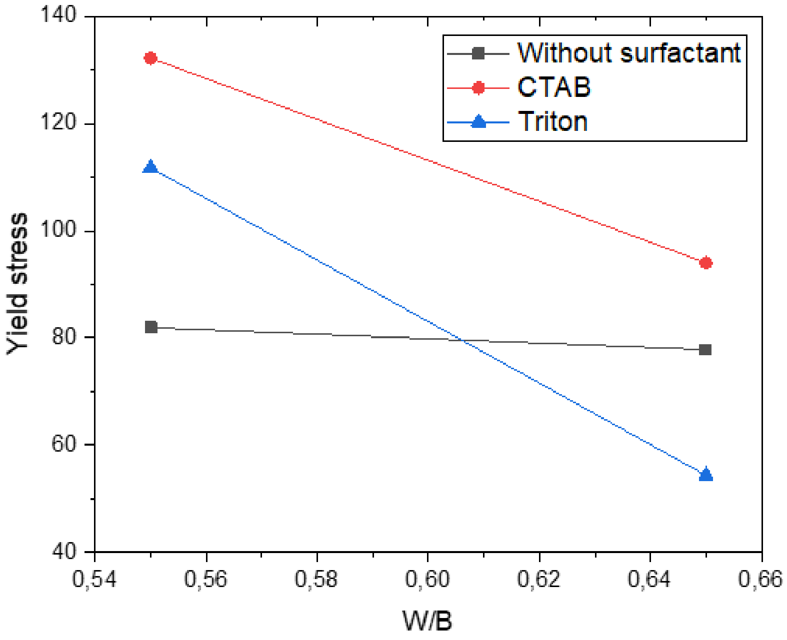


Fig 15. Effect of W/B ratio on the yield stress NaOH:5, H_2O_2:0%)

6.1.9. Effect of molarity on the yield stress:

In figure 16, the effect of molarity on yield stress is shown. The value for yield stress decreased dramatically for CTAB by increasing the molarity. The effect on yield stress for triton is also inversely proportional and decreasing with the increasing the molarity of the mixture. The behaviour of the reference sample and the Triton is almost same, and the same decreasing trend can be seen in the both case

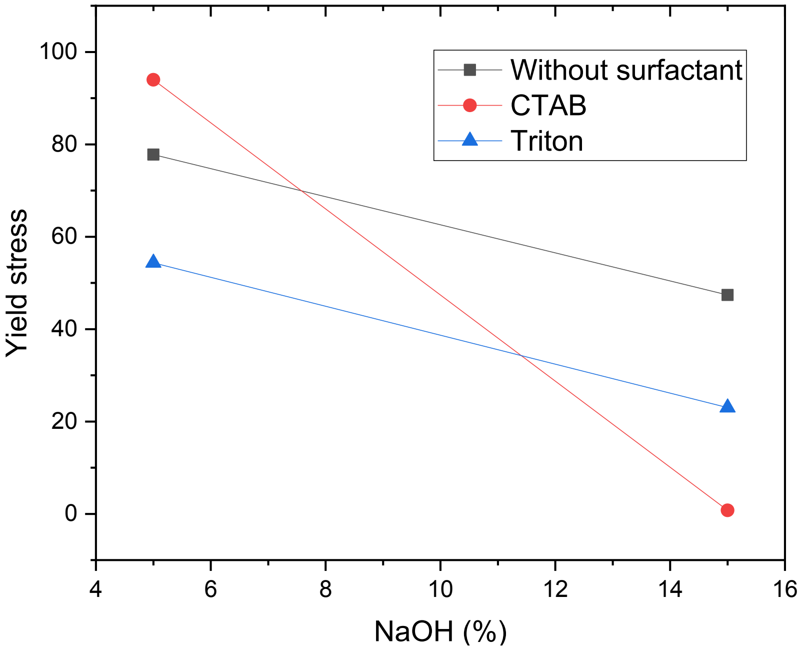


Fig 16. Effect of molarity of NaOH on yield stress (W/B:0.65, H_2O_2:0%)

6.2. Hardened Properties:

The following table 3, shows the harden properties and the standard deviation value for each property. The standard deviation was calculated on the average of three specimens. The result is also shown in the graphical form to explain the effect of H_2O_2 and effect of molarity on the harden properties. 36 mixes were tested to analyze the harden properties and made a comparison on different parameters identified.

Table 3: shows the data gathered for harden properties

Mix number	strength (MPa)	S.D	water absorption (%).	S.D	Density (kg/m$_3$)	S.D
5N5.5W2	7.76	0.77	38.24	0.43	1036.73	16.92
5N5.5W2T	8.68	0.62	37.78	0.30	1067.71	6.77
5N5.5W2C	9.28	0.38	37.19	0.07	1076.07	24.97
5N5.5W1	9.93	0.12	37.09	0.09	1084.28	28.10
5N5.5W1T	9.96	0.62	37.03	0.18	1132.65	11.65
5N5.5W1C	10.15	0.56	36.87	0.22	1150.87	6.63
5N5.5W0.5	11.12	0.06	36.58	0.14	1160.78	14.16
5N5.5W0.5T	11.13	0.88	36.32	0.39	1166.98	17.41
5N5.5W0.5C	11.22	1.07	36.27	0.61	1193.42	23.46
5N5.5W0C	12.70	0.71	35.47	0.50	1209.18	26.17
5N5.5W0T	13.32	2.07	34.78	1.20	1249.17	104.95
5N5.5W0	14.11	2.08	34.25	1.13	1272.56	104.05
10N5.5W2	10.20	0.70	37.01	0.76	1039.17	25.31
10N5.5W2T	10.92	0.42	35.90	0.47	1067.20	31.68
10N5.5W2C	11.60	0.30	35.16	0.21	1101.06	51.52
10N5.5W1	11.67	1.79	34.77	0.29	1144.59	42.71
10N5.5W1T	12.16	1.72	34.67	0.28	1225.42	15.39
10N5.5W1C	14.99	0.91	34.11	0.10	1242.52	27.41
10N5.5W0.5	15.28	1.57	34.07	0.57	1263.06	27.42
10N5.5W0.5T	16.70	1.20	33.88	1.58	1308.14	19.70
10N5.5W0.5C	18.41	0.57	32.78	1.55	1328.70	19.24
10N5.5W0C	19.01	0.50	30.12	0.87	1356.23	17.96
10N5.5W0T	19.56	7.30	29.11	3.07	1375.60	18.04
10N5.5W0	20.00	6.87	28.00	3.26	1400.12	17.89
15N5.5W2	7.14	1.59	35.00	0.20	1005.60	52.89
15N5.5W2T	9.39	0.55	34.83	0.26	1037.55	43.80
15N5.5W2C	10.20	1.21	34.51	0.32	1130.30	24.29
15N5.5W1	10.43	1.16	34.20	0.24	1130.61	31.71
15N5.5W1T	12.41	0.30	33.73	0.17	1181.97	18.50
15N5.5W1C	12.47	0.42	33.66	0.52	1206.74	10.05
15N5.5W0.5	12.95	0.75	33.33	0.56	1227.21	0.79
15N5.5W0.5T	13.31	2.73	32.43	0.28	1228.83	5.36
15N5.5W0.5C	14.40	2.28	31.98	0.18	1228.93	5.72
15N5.5W0C	18.49	0.47	31.74	0.81	1240.25	6.41
15N5.5W0T	18.19	0.65	31.55	0.80	1241.72	6.40
15N5.5W0	19.11	1.57	29.94	1.22	1254.53	10.62

6.2.1. Effect of H_2O_2 on compressive strength and density:

In the figure 17: The effect of H_2O_2 on compressive strength is illustrated. There were three variation in the specimens studied, i.e., without surfactant, CTAB and Triton. The percentage of H_2O_2 were in the range of 0,0.5,1,2 %. The decreasing trend of strength with increasing aerating agent can be seen in each case, but the effect is higher for specimens without surfactant. The trend for the CTAB and triton is quite same, and CTAB performs better for any given percentage of H_2O_2. The density of all the three kind of samples decreased with the increase of H_2O_2 same as compressive strength. And hence, density is directly proportional to the compressive strength as a rule.

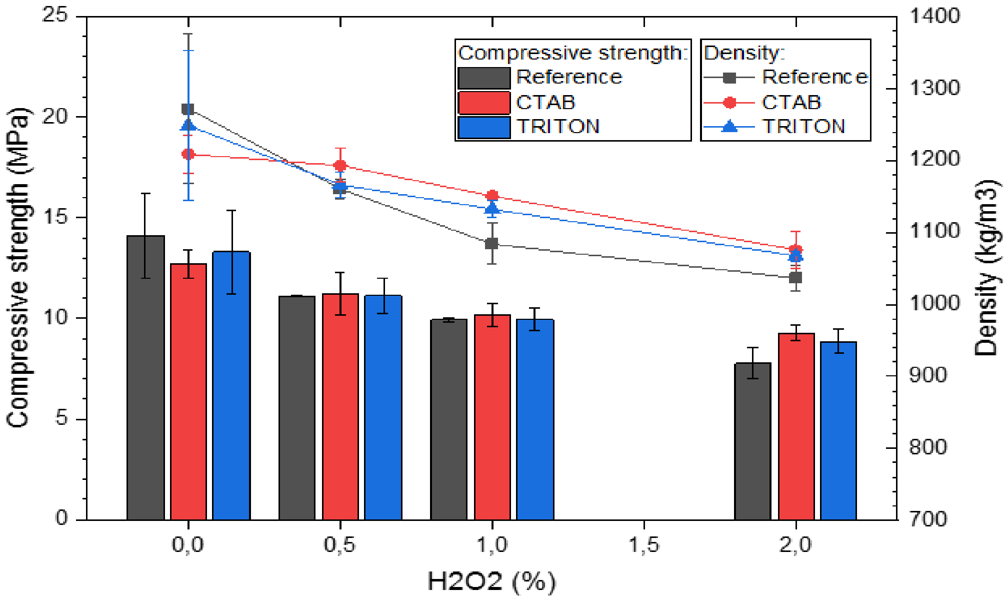


Fig17. Effect of H_2O_2 on compressive strength and Density (W/B:0.55, NaOH:5)

6.2.2. Effect of Molarity on compressive strength and density:

Figure 18: shown the effect of molarity on the compressive strength of the foam concrete. Samples were categorized into three different categories: without surfactant (Reference), CTAB and Triton. The compressive strength for each sample increased by increasing the molarity of NaOH. The compressive strength increased dramatically for the specimen with surfactants (CTAB/Triton) with the increase in molarity. Corresponding dry density also increased with the strength value. The standard deviation was higher for the reference sample with 15 % NaOH and for CTAB sample with 5% NaOH.

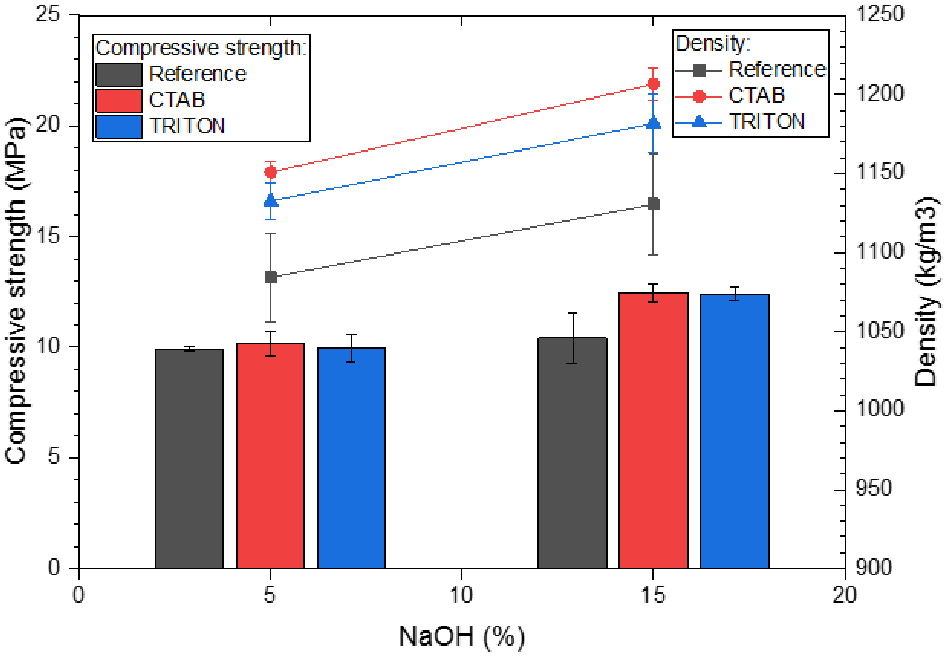


Fig 18. Effect of molarity on compressive strength and Density (H_2O_2:1%,W/B:0.55)

6.2.3. Effect of H_2O_2 on water absorption and density:

The following figure 19, gives the insight on the effect of H_2O_2 on water absorption of foam concrete samples. The effect on the water absorption was opposite to the compressive strength. The water absorption for each sample increased by increasing the percentage of H_2O_2 but the increment in value were very small between 1 and 2%. The reason for the increment in the water absorption is due to increase in the number of pores with higher dosage of H_2O_2.With the increase of percentage of hydrogen peroxide the number of pores also increased. The standard deviation for reference and Triton specimens were notables compared to CTAB, indicating CTAB gives better uniformity in distribution of pores all over the mix. The density and water absorption are inversely proportional to each other as one is increasing and other is decreasing as shown in the figure 19.

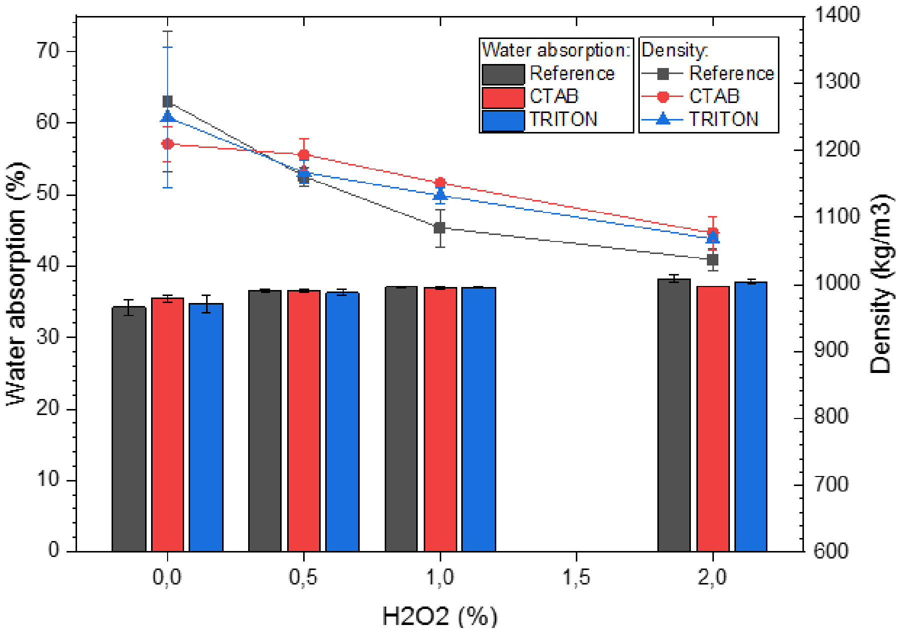


Fig 19. Effect of H_2O_2 on water absorption and density. (W/B:0.55, NaOH:5)

6.2.4. Effect of Molarity on water absorption and to density:

Figure 20 gives the clear picture for the effect of molarity on water absorption and shows the comparison between water absorption and density. The water absorption decreases with increasing the molarity from 5M to 15M for each of the mix including reference, CTAB and Triton. The reason of the low water absorption with high molarity is the formation of dense microstructure which lowered the number of pores and their cross-sectional area. The trend of density is inversely proportional to water absorption with the increase of molarity. water absorption totally depends on the size, shape and interconnectivity of the pores of geopolymers. Gorhan and Kurklu gave the concept that, the water absorption decreases with increasing the curing time when they used curing temperature of 85 °C. They also found that the increase in molarity also decrease the water absorption [76]. Djobo et al. investigated the effect of curing temperature and by increasing the curing temperature the water absorption also increased and the variation in water absorption before and after 28 days of curing was not remarkable [77]. Yliniemi et al. studied the effect of the introduction of light weight clay aggregate in the specimens and found that the water absorption increased because of the addition of light weight aggregate as compared to those with only traditional aggregates [78]

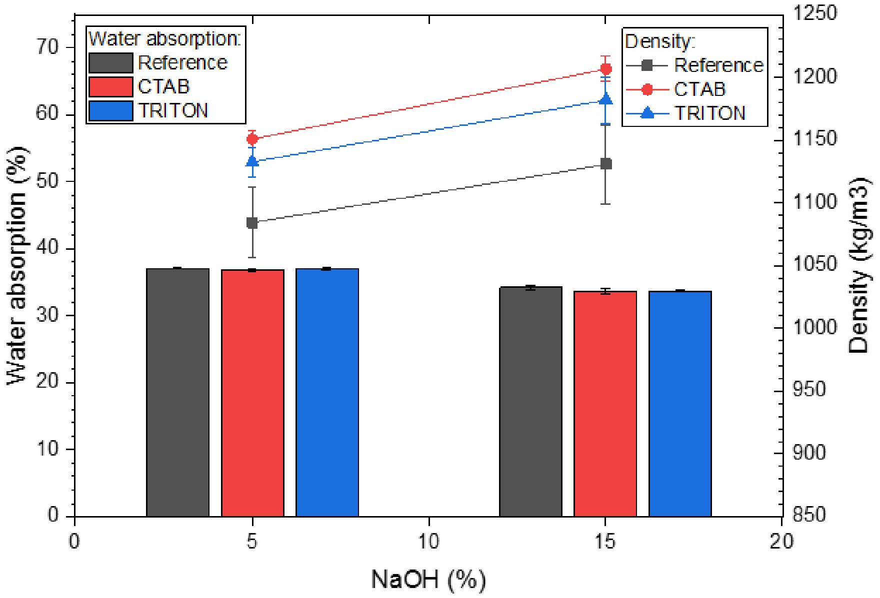


Fig 20. Effect of molarity on the water absorption and density (W/B:0.55, H_2O_2:1%)

6.3.2. XRD results of our study:

Figure 21: shows the XRD results for the raw material before the formation of samples of different molarity. The result shows only two phases in it which are quartz and muscovite (hydrated phyllosilicate). In the figure 22, the XRD results of three samples after alkali activation of impure kaolinite with NaOH of different molarity (5N,10N, 15N) are shown. The number of phases shown in the XRD curve after activation are five. The x axis showing the angle of diffraction and the y-axis showing the light intensity which is hitting the sample. The XRD results revealed that, the material contained quartz, muscovite, sodalite, nepheline and Kaolinite. The amount of quartz was larger and on second number after kaolinite. Kaolinite was the main fraction for our focus of study. The analysis shows that, the phases are quite similar in all three samples with 5N,10N and 15 N of NaOH.

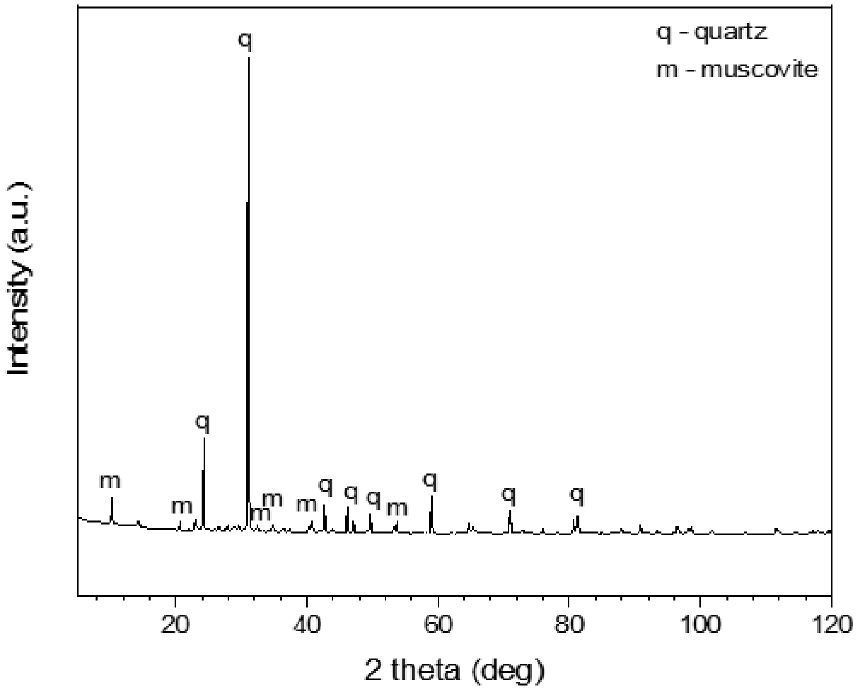


Fig 21. XRD analysis of impure kaolinite

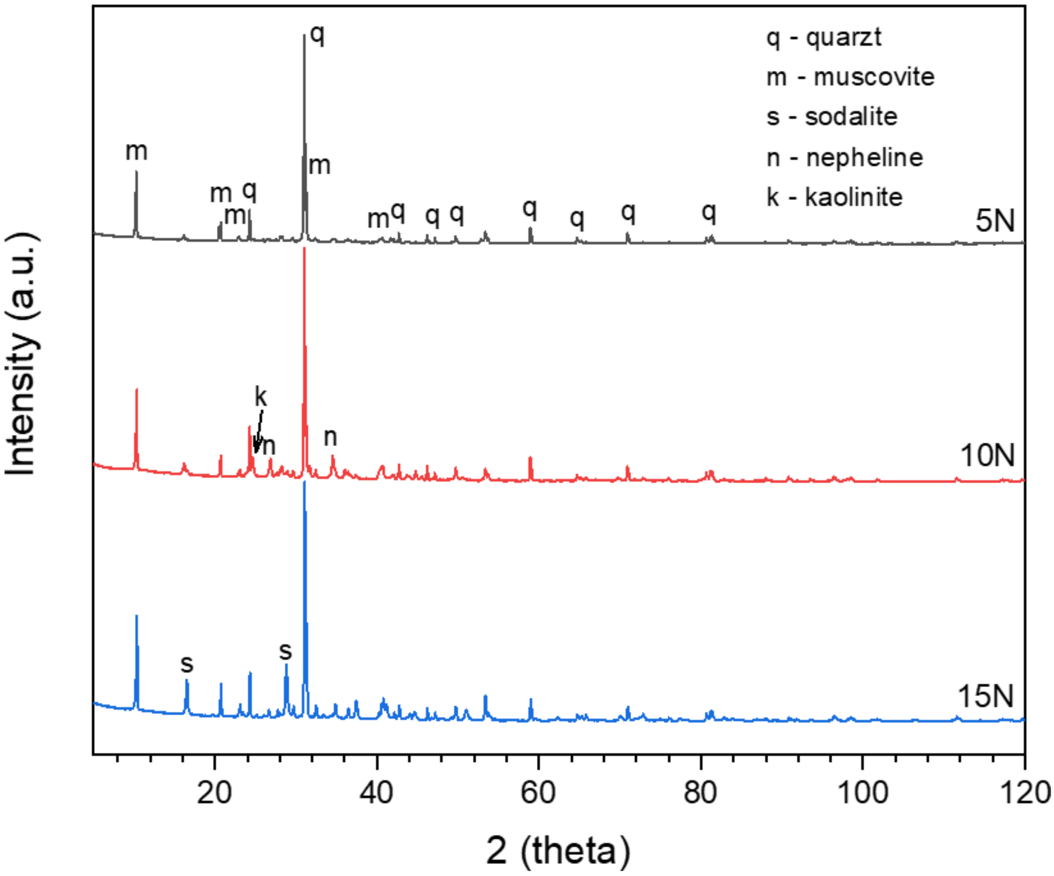


Fig 22. XRD analysis of alkali activated impure kaolinite with 5N,10N,15N NaOH

6.3.3. FTIR Analysis of study:

FTIR results shown below in the figure 23: carried out in the range of 500 to 4000 cm-1 wave number. The samples of the study were same as used for XRD. FTIR basically give insight about the structural formation of polymers during their activation. It can be seen in the graph that, the result for all three samples 5N,10,15N is quite same and showing the similar trend. The clear difference in the peaks of different sample on wave number 3500,1500 and 500.The x axis shows the wavenumber of light and the y axis show the absorbance which depend on the excitement of the molecules of material.

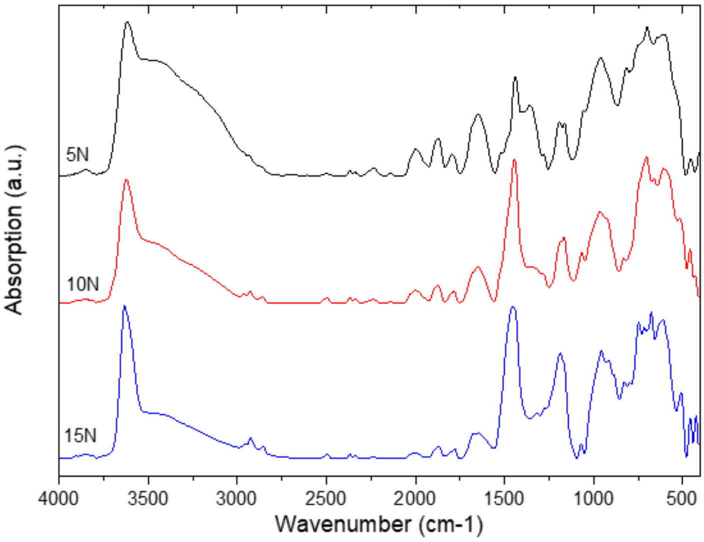


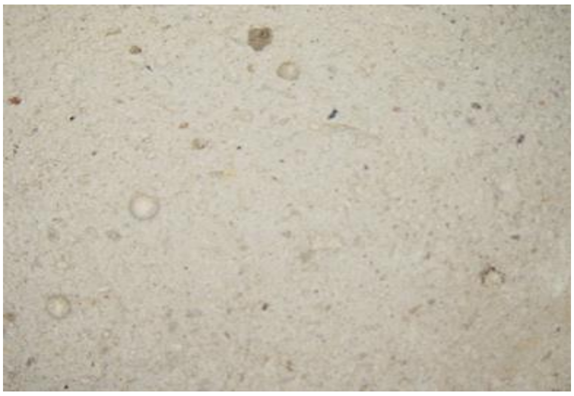
Fig 23. FTIR analysis graph

6.4. Microscopic analysis:

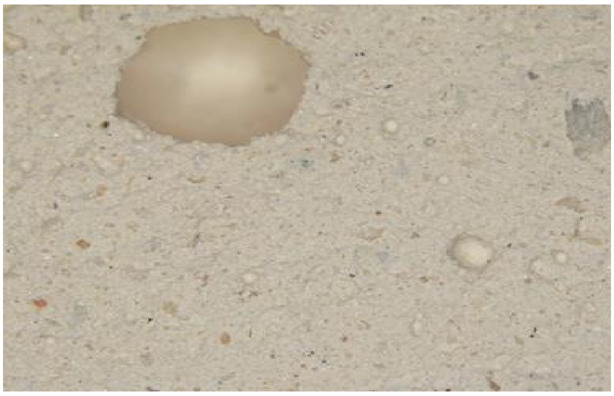
Microscopic study was carried out by using Olympus polarizing microscope BX51P with micropublisher 5.0 RTV camera. Microscopic analysis was done to compare the pore structure with different percentage of H_2O_2 and molarity of NaOH. Number of samples tested were twelve which is shown in the following figures 24 and 25.

In the figure 24, the comparison made between 5% and 10% molarity to show the effect of molarity on the pore size and structure. The number of pores increased with increasing molarity of the NaOH used for activation. The pore size is also bigger in the 10 N samples. The pores with CTAB and Triton are in the regular shape and smaller in the size as compared to the pores shown in the reference samples. The reason behind it that, the surfactants control the pores chemistry and help in getting good shape discussed already in the surfactants section.

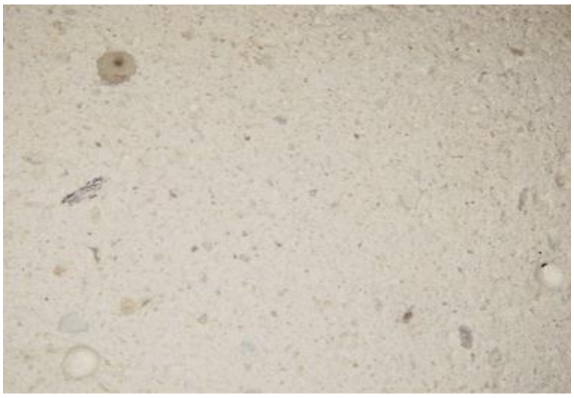
It can be seen in the figure 25: that the pore size with 2% of H_2O_2 is large and the number of pores is also more in that case. Without surfactant or reference the pore structure is irregular in shape while in other sample with surfactant the pore structure is regular in shape. There is not very big difference in the number of pores in the case of reference and CTAB for both 1% and 2%. A clear contrast can be seen in the case of the triton and the number of pores with 2% is much more as compared to 1%. The pore structure is very clear and regular in the case of Triton. The pore size is in medium size in the sample of Triton.



5N5.5W1



10N5.5W1



5N5.5W1 CTAB

10N5.5W1 CTAB

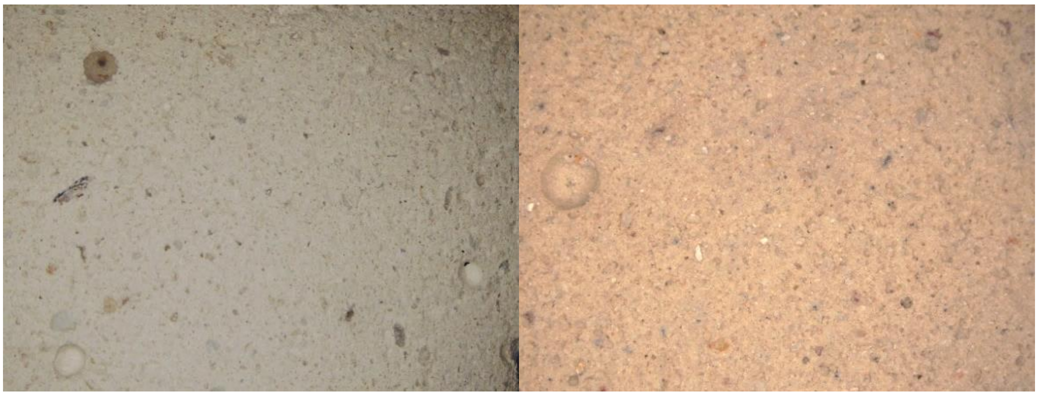
5N5.5W1 Triton

10N5.5W1 Triton

Fig 24. X12 magnification, Scale 10 mm: Effect of molarity on the pore size and shape.

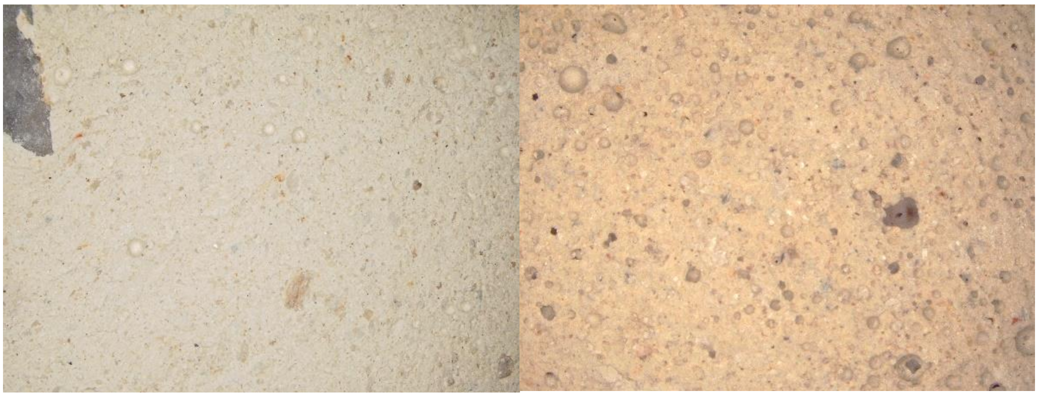
5N5.5W1

5N5.5W2



5N5.5W1 CTAB

5N5.5W2 CTAB



5N5.5W1 Triton 5N5.5W2 Triton

Fig 25. X12 magnification, Scale 10 mm: Pore structure analysis of the sample by polarizing microscope

7. Conclusion:

The conclusion of the thesis can be summarized in the way that, the purpose of the study was to determine the behavior of the impure kaolinite samples and use them as a base material to produce porous concrete to be used in the construction industry.

The fresh density and aeration rate were affected by different factors including percentage of H_2O_2, Molarity and W/B ratio of the samples.

The workability was well maintained with lower molarity and with high-water binder ratio. However, the mix was workable at higher molarity of NaOH with the range of 10 and 15 molar solution.

The compressive strength was maximum for the mix without any aerating agent activated with 10M NaOH solution with the w/b ratio of 0.55, which was 20 MPa. It reduced up to almost 50% when aerating agent has been added.

The minimum water absorption of 29.94% achieved with 15M NaOH solution with w/b ratio of 0.55.

Fine and evenly distributed pores were found in specimens with Triton surfactant, however the specimens made with CTAB performed better when cured at higher temperature (400 ˚C).

Use of surfactants affects both fresh and hardened properties of foamed alkali activated material. However, it is also possible to make foam without surfactants. CTAB helps in producing foam with better strength and gives better resistance at high temperature curing whereas Triton is good in even pore distribution which helps in better functional properties.

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