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Effect of mixing conditions on the density, morphology, thermal and mechanical properties of mineral foam

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Abstract

Mineral foam concretes are classified as lightweight concretes, which are characterized by the presence of air voids in the materials due to the use of a suitable foaming agent. Their physical and mechanical properties are strongly governed by microstructural properties, which depend on several factors such as foam quantity, water/binder ratio (W/B), and the mixing process. In this study, mineral foams were prepared with a mixed foaming method. Gypsum is used as a binder, flax shives as vegetative aggregates, and Betomouss as the foaming agent (sodium lauryl ether sulfate as the main tensioactive molecule). The impact of mixing conditions (stirring rate, stirring time, and W/B ratio) on the performances of foams is investigated. The results show that varying these mixing conditions will induce high variation of foam performances at the fresh and hardened states. Furthermore, increasing the stirring rate and stirring time generates an increase of the air-void system, finally causing materials to decrease foam density. Consequently, this entails an improvement in thermal conductivity and a reduction of mechanical properties. In addition, increasing the W/B ratio leads to an increase in spreadability, density, and mechanical properties. In contrast, foams with a low W/B ratio have the best thermal properties.

Keywords: Mineral foam; Mixing conditions; Gypsum; Flax shives; Density; Thermal conductivity; Mechanical properties.

1. Introduction

In Europe, the building sector is considered as one of the sectors that most impacts the planet's environment and is the principal source of energy consumption, accounting for approximately 40% of global energy demands. The building sector is responsible for 36% of greenhouse emissions, mainly due to heating, cooling, and air conditioning systems [1,2]. Therefore, the preservation of the planet is becoming a major strategic issue in all regions of the world. Consequently, new strategies are being developed in Europe in favor of reducing CO₂ emissions and saving energy. These strategies aim to ensure environmental protection by facilitating the advent of more innovation, resource-efficiency, and competition to encourage the use of renewable resources for industrial purposes. In this context, awareness of the preservation of the planet has motivated researchers and manufacturers in the building sector to increase their integration of insulation materials in building envelope systems. The most commonly used insulation materials in the building sector are polyurethane foam, fiberglass, mineral wool and polystyrene [3]. These conventional materials are always associated with a load-bearing structure due to their restricted mechanical performances [4,5]. However, some insulation materials such as foam concrete have been developed, both to play a partial load-bearing role as well as to simultaneously provide thermal insulation performances [6]. These insulation materials can be an alternative to conventional insulation materials [7,8]. Foam concrete, also known as lightweight concrete, is composed of cement-based slurry, water, stable foam, and possibly aggregates. Foam concrete could be produced using either chemical or mechanical methods. The chemical method involves inserting an expansive agent (e.g., aluminum powder) into a mineral matrix [9,10]. Indeed, when the expansive agent is in contact with water and hydroxides in the alkaline medium, hydrogen bubbles are released, and hydrolyzed metal complexes are created. The most common product obtained by this method is autoclaved aerated concrete [11]. The chemical method is not environmentally friendly compared to the mechanical method as it is based on the use of foaming agents (also called surfactants) to produce compact mineral foam [12]. Two mechanical methods can

be used to produce mineral foams, namely pre-foaming and mixed foaming [6,13]. The pre-foaming method consists in separately producing mineral paste and stable performed aqueous foam (i.e., where the foam maintains its initial properties while being generated) and then thoroughly blending the foam into a mineral paste. In the mix-foaming method, the surfactants are added within the mineral suspension, which allows air voids to be entrapped in the solid components during mixing. The presence of air voids generates various specific properties of lightweight concrete compared to normal-weight concrete, e.g., low self-weight, thermal insulation, and acoustic absorption [12]. However, many parameters such as the paste intrinsic properties, fabrication methods, components (e.g., W/B, foaming agent type and size of filler), and curing conditions can impact the performances of lightweight concrete either at the fresh or at hardened state [13]. Liu et al. [14] revealed that varying the W/B ratio induces a change in the performance of foam concrete. A high W/B ratio results in lower relative viscosity and weak bubble-maintaining capacity of foam concrete. Nambiar & Ramamurthy [15] highlighted the impact of the type of filler materials used for foam concrete production on several properties. In fact, the finer filler leads to more uniform air-void distributions in foam, resulting in an increase of compressive strength. Moreover, Kearsley & Wainwright [16] have reported that the compressive strength of foam concrete decreases exponentially with a diminution in density. The air void distribution is the most crucial property influencing the mechanical performances of foam concrete [17,18]. In addition, this variation of foam density has a direct influence on thermal properties. Many authors [6,19,20] showed that the thermal conductivity varies linearly with the density, ranging between 0.1 and 0.77 W/m.K for dry densities of 600–1850 kg/m³. Besides the performances of lightweight mineral foam, many studies in the literature were focused on using cement for manufacturing foam concrete. However, cement materials require a large quantity of energy to be produced, which greatly impacts the environment. Consequently, gypsum can be used to produce mineral foam since it is more eco-friendly than cement. In addition, gypsum has several advantages such as excellent fire-protecting and heat-resistant properties, good workability, and acoustic insulation and humidity controlling abilities, to name but a few [6,12,21,22]. In this sense,

several researchers have explored the possibilities of using gypsum as a binder. Vimmrová et al. [23] developed foam concrete using gypsum as a binder, micro silica as a filler, and aluminum sulfate powder as an expansive agent. The authors demonstrated that the chemical method provides poor mechanical properties due to the presence of a big bubble. Samson et al. [6] developed mineral foams composed of gypsum (90%) and Portland cement (10%), mixed with a low W/B ratio (0.35). The authors showed that gypsum–cement foam performances can be enough to generate partial load-bearing insulation products. Recently, Capasso et al. [24], studied an innovative method that consisted in adding different amounts of pre-manufactured foam (5, 7.5, and 10% wt) to gypsum pastes. These authors show that increasing foaming content leads to improving thermal properties, a decrease of flexural strength, and modification of the compressive behavior, owing to the increase of porosity. To further reduce the environmental impact of foam concrete, the waste by-products/vegetable co-products (e.g., flax, hemp, and miscanthus) can be used as bio-aggregates [12,25]. Beyond their sustainability and environmental protection advantages, these bio-aggregates have a low density, low thermal conductivity, and high hygroscopic properties. Thus, adding this bio-aggregate can be an appropriate method to increase the porosity in order to enhance the physical and insulation characteristic of mineral foam composite.

Although many studies have been performed on mineral foam concrete, one crucial factor in foam development that has not been given sufficient attention is the mixing process. The manufacturing process depends on mixing conditions. Therefore, the impact of mixing conditions on the performances (mechanical or physical) needs to be studied and understood.

The purpose of the present work is to study the effect of the mixing conditions (i.e., stirring rate and stirring time) on the performances of mineral foam and compare the difference between mineral foams performed with two different W/G ratios. Gypsum is used as a binder, flax shives have been chosen as aggregates and the foaming agent employed to create air voids in the mixtures is Betomouss®.

Different properties of mineral foams were evaluated at the fresh and the hardened state such as density, spreadability, morphology, thermal conductivity, and mechanical properties (compressive and flexural strength).

2. Materials and method

2.1. Raw materials

The mineral foams prepared in this study are composed of gypsum paste with flax shives aggregates, citric acid, and foaming agents. The gypsum is used as a binder. It is commonly called Paris plaster; it contains beta hemihydrate ($\text{CaSO}_4, 1/2\text{H}_2\text{O}$) and anhydrite (CaSO_4).

The vegetative aggregates used are flax shives that were provided in bulk by the flax scutching company Texilis (France). The flax shives are the wooden part of the flax stalk, broken into pieces and separated from the fiber during the breaking and scutching processes. The flax shives have variable sizes from 1 to 33 mm (Figure.1), and their bulk density is around $105 \text{ kg}\cdot\text{m}^{-3}$. The foaming agent employed to create air voids in the mixtures was generated using Betomouss® purchased from Sika, commonly used for concrete foam, including sodium lauryl ether sulfate as a tensioactive molecule. Citric acid monohydrate (CAM), supplied by “BFC SAS” from Rémalard-en-Perche (FR61110), was previously crushed and sieved at $250 \mu\text{m}$ before being used as the setting retarder.

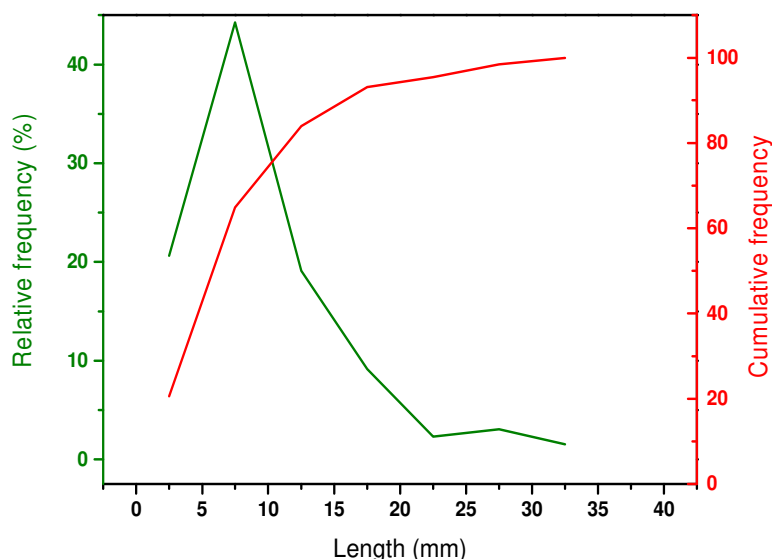


Figure.1. Characterization of flax shives length by image processing

2.2. Material processing

The mineral foams were prepared with a new mixing method which consists in progressively adding different foam components during mixing (Figure.2). A mortar mixer (Perrier type) with a bowl of five liters is used to produce flax foam materials. Different formulations with a change of mechanical mixer parameters were tested (Tables 1 and 2). The mass proportion of foam components is fixed for all formulations, except for water. Indeed, the flax shives/ gypsum ratio (F/G), foaming agent/gypsum ratio (FG/G) and citric acid /gypsum ratio (AC/G) are 0.2%, 0.77%, and 0.02%, respectively. Two water/gypsum ratios W/B (0.77 and 1.12), are tested under a change of mechanical mixer parameters. A similar protocol is followed for all formulations to introduce components to the mixer. Firstly, to homogenize the mixture, gypsum and citric acid are introduced in the bowl and mixed for 20 seconds with an initial stirring rate of 100 rpm. Then, water mixed to the foaming agent is added to the mixture progressively for 20 seconds by keeping 100 rpm as stirring rate. It should be emphasized that this initial stirring rate was fixed for the homogenization step before applying a specific stirring rate and time for each formulation. The mixing continued for 20 seconds with a specific stirring rate selected for each formulation. Then a slowdown to the initial stirring rate for 90 seconds was performed to introduce flax shives progressively to the mixture. Finally, a last mixing step was applied using the selected stirring rate and time (Table 2) to obtain the final foam product. Once the mixer is stopped, the density and spreadability of the foam are measured. The foam is cast into molds for tests in the hardened state. Samples are kept and stored in their mold until hardening in a climatic room, which maintains the controlled temperature at 20°C and relative humidity at 50%. The performance test at the hardened state was performed after a curing time of 60 days.

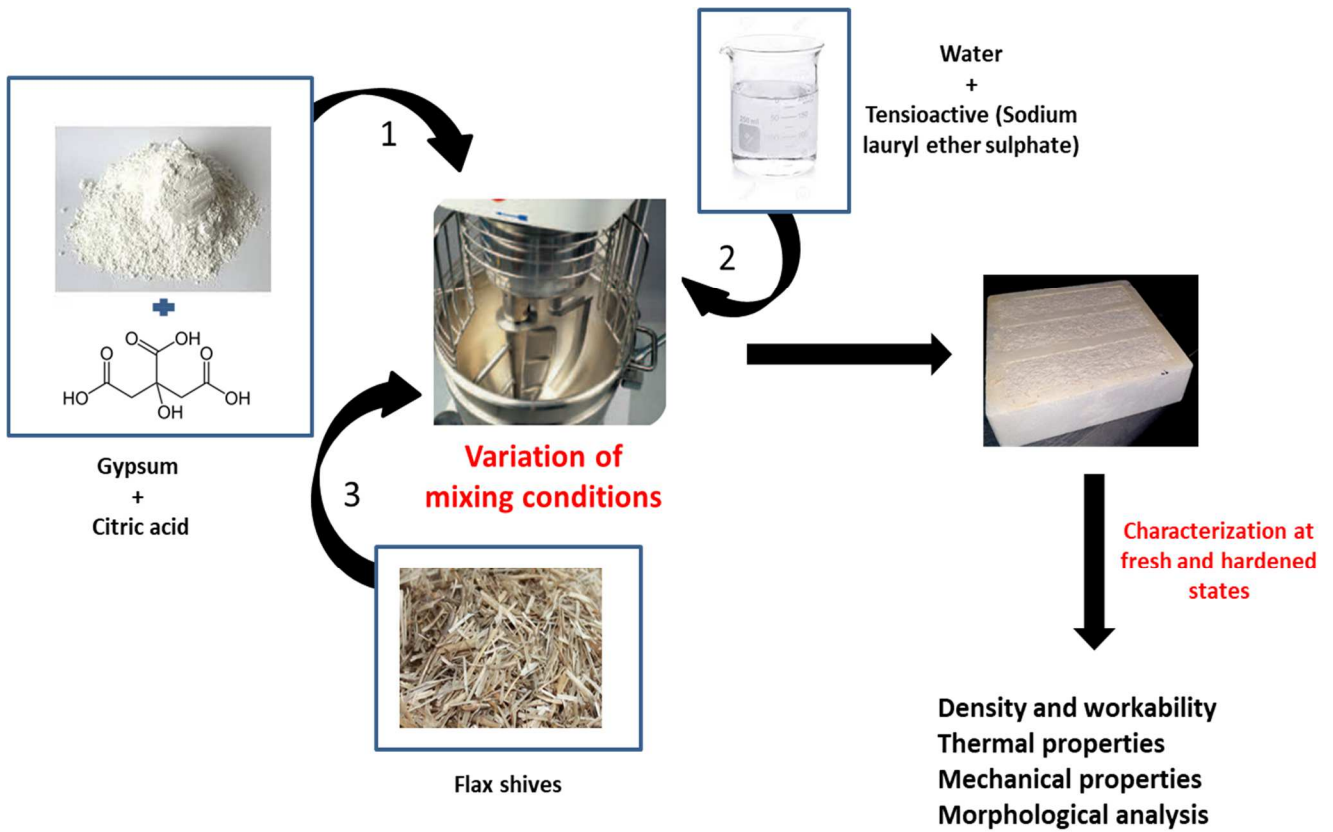


Figure.2. Mineral foam processing steps

Table.1. Mix proportions of raw materials

(F/G)	(FG/G) %	(AC/G) %	(W/G)
0.20	0.77	0.02	0.77
0.20	0.77	0.02	1.12

Table.2. Mechanical mixer parameters performed during foam preparation

Stirring rate (rpm)	Stirring time (s)
100	120
150	120
200	120
250	120
300	120
200	60
200	180
200	300

2.3. Characteristics Methods

2.3.1. Fresh density and spreadability

After the mixing step, the mineral foam's fresh state characteristics (fresh density and spreadability) were investigated. Firstly, the fresh density of the samples was calculated using the standard mass over volume formula. The mixed material was poured into a pre-prepared $\text{Ø } 50 \times 50$ cylindrical plastic. The density was determined by weighting the foam sample and measuring the weights and volumes of the molds. The spreadability of foam concrete was determined by the flow table test [26], which was made by filling the mix in two layers of the open-ended cylinder (mold). To ensure a uniform filling of the open-ended cylinder, sufficient tamping pressure was applied. Then the mold was lifted vertically, and the fresh mineral foam was spread on the metallic plate. Each spreading diameter was measured after the application of 15 shocks. The values of spread diameter were obtained by the average measurement of diameter distribution in four orthogonal directions.

2.3.2. Morphological analysis

The morphology of different samples was observed through an optical microscope DigiMicro 2.0 (DigiMicro Skale microscope, DNT, Germany), and the software used was MicroCapture.

2.3.3. Thermal properties

Thermal conductivity represents the ability of heat to spread in a material for a given temperature gradient. To determine the thermal conductivity properties of the samples, the FP2C conductivity meter (NeoTIM®, France) was used under stable humidity and temperature environment. The thermal conductivity was measured with a hot linear wire sensor (50 mm) following the ASTM D5930-97 standard. This method consists in placing a heating wire between the surfaces of two identical samples of the material to be characterized. Then a constant heat flow is applied to the sensor and the temperature change of the wire is examined.

Thermal tests on mineral foam specimens were conducted after 60 days of setting. To ensure good repeatability of results, at least five tests were performed for each formulation.

The results were analyzed statistically using the Analysis of variance ANOVA and a Tukey multiple comparison test at the significant level of 5%.

2.3.4. Mechanical properties

Mechanical tests were performed on prismatic specimens ($160 \times 40 \times 40 \text{ mm}^3$) and cubic specimens ($40 \times 40 \times 40 \text{ mm}^3$) for flexural test and compressive tests, respectively, using an electromechanical testing machine with a force capacity of 50 kN . This equipment ensures a satisfactory level of precision ($\pm 0.5\%$ between 0 and 0.5 kN and $\pm 0.3\%$ between 0.5 kN and 50 kN) for the non-bearing products. The three-point flexural test was carried out with a crosshead rate of 1 mm/min at room temperature. The specimens were placed between two supports at a distance of 120 mm according to the EN 12390-5 standard [26] adapted for concrete foams.

The compression test was performed on the same testing machine at room temperature with a crosshead rate of 1 mm/min according to NF EN 679 standard [27]. At least five specimens were tested to evaluate the reproducibility. Differences between obtained results were evaluated using the Turkey multiple comparison test with a 5% level of significance.

3. Results and discussion

3.1. Effect of stirring rate and time on fresh mineral foam suspension density

The influence of mixing conditions on the fresh density is shown in Figure.3, which demonstrates the fresh densities of mineral foams do change with varying stirring rates and stirring times. Increasing of stirring rate of the mixer leads to a decrease in the density of the foam. For foam prepared with a W/G ratio of 0.77, the densities are 444, 373, 341, 368 and 355 kg/m³ for 100, 150, 200, 250 and 300 rpm, respectively. Regarding foams with a W/G ratio of 1.12, the densities are 625, 596, 532, 482 and 265 kg/m³ for 100, 150, 200, 250 and 300 rpm, respectively.

A similar tendency could also be observed during the change of stirring time. A high stirring time induces a low foam density. For foam prepared with a W/G ratio of 0.77, the densities change from 417, 341, 363 and 262 kg/m³ for 60, 120, 180 and 300 s, respectively. As regards foams with a W/G ratio of 1.12, the densities are 599, 532, 412 and 244 kg/m³ for 60, 120, 180 and 300 s, respectively. This diminution of foam density with increasing stirring rate and stirring time is related to the increase of the foaming capacity, air-void diameter, and porosity of foams. It is well known that increasing porosity induces a decrease in foam density [17,28]. High stirring rate and stirring time lead to an increase of pore numbers and foam volumes, whereby causing a reduction of the density of the final material.

In addition, the results also show that according to the W/G ratio added during foam preparation, the density changes. In this respect, mineral foams prepared with a low W/G ratio are less dense compared with foams prepared with a high W/G ratio, except when stirring rate and stirring time are high e.g., 300 rpm and 300 s. At a higher W/G ratio, the foam densities are high since increasing water contents make the gypsum slurry too thin to hold the bubbles conducting to a segregation of the foam from the mix. However, a high increase in stirring rate or stirring time leads to having more air-void and large bubbles resulting in a reduction of foam density [17,29].

According to these results, it can be concluded that stirring rate, stirring time and W/G are three key variable parameters that govern the variation of fresh density of mineral foam.

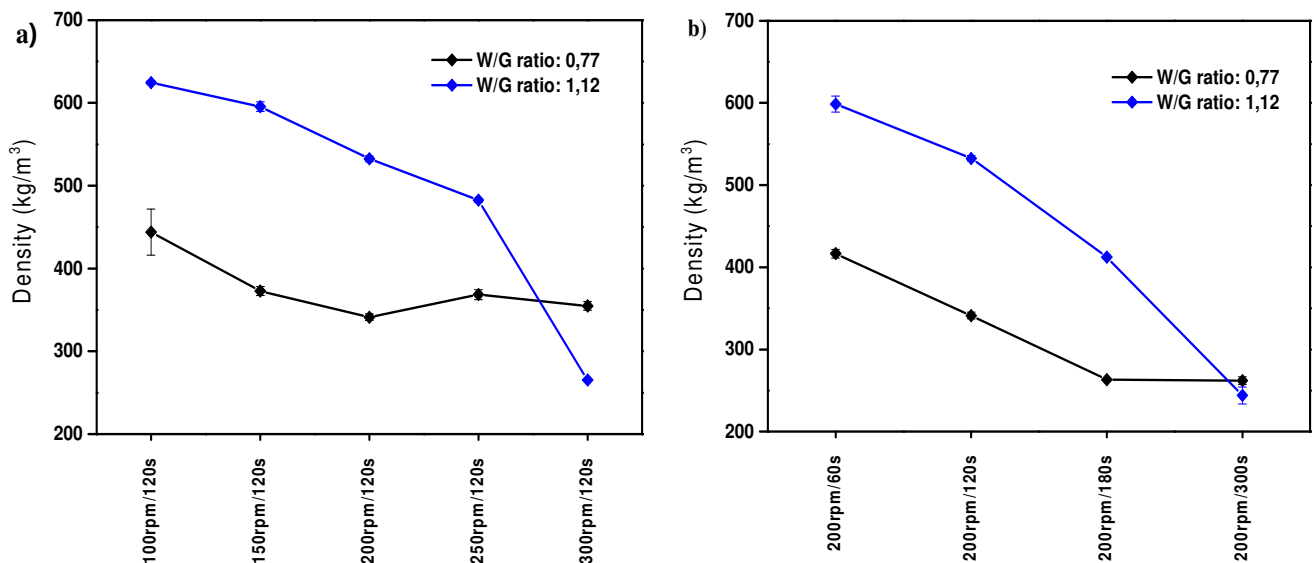


Figure.3. The density of foam as a function of a) stirring rate and b) stirring time for different W/G ratios

3.2. Effect of stirring rate and time on the spreadability of fresh mineral foams

The high fluidity of the fresh mineral foam is an essential criterion in industrial use, mainly for casting foam with low density. For this reason, the behavior of mineral foam at the fresh state is studied using the flow table test. The test aims to follow the evolution of the spread diameter of mineral foam after changing the stirring time, stirring rate, and water quantity.

Figure.4 shows that the spread diameter of foams slightly changes when varying stirring rates and stirring times. Contrariwise, as expected, varying W/G induces a significant variation in the foams' spread diameter due to changes in the surface area of solids [30].

For foam prepared with a W/G ratio of 0.77, the spread diameters are 154, 185, 183, 174 and 172 mm for 100, 150, 200, 250 and 300 rpm, respectively. As regards foams with a W/G ratio of 1.12, the slump values are 178, 193, 194, 193 and 184 mm for 100, 150, 200, 250 and 300 rpm, respectively.

In general, increasing stirring rate or stirring time allows to increase the spread diameter at the beginning, but a prolonged stirring rate or stirring time induces a reduction of foam spread diameter.

This could be explained by foam structure change. Low spread diameters are observed at low stirring rates or stirring times, as mineral flax foams are denser.

The reduction of spreadability at a high stirring rate or stirring time could be related to higher air content and decrease of self-weight of mineral foam. The adhesion between the bubbles and solid particles in the mixture increases the rigidity of the mix, reducing the spreadability.

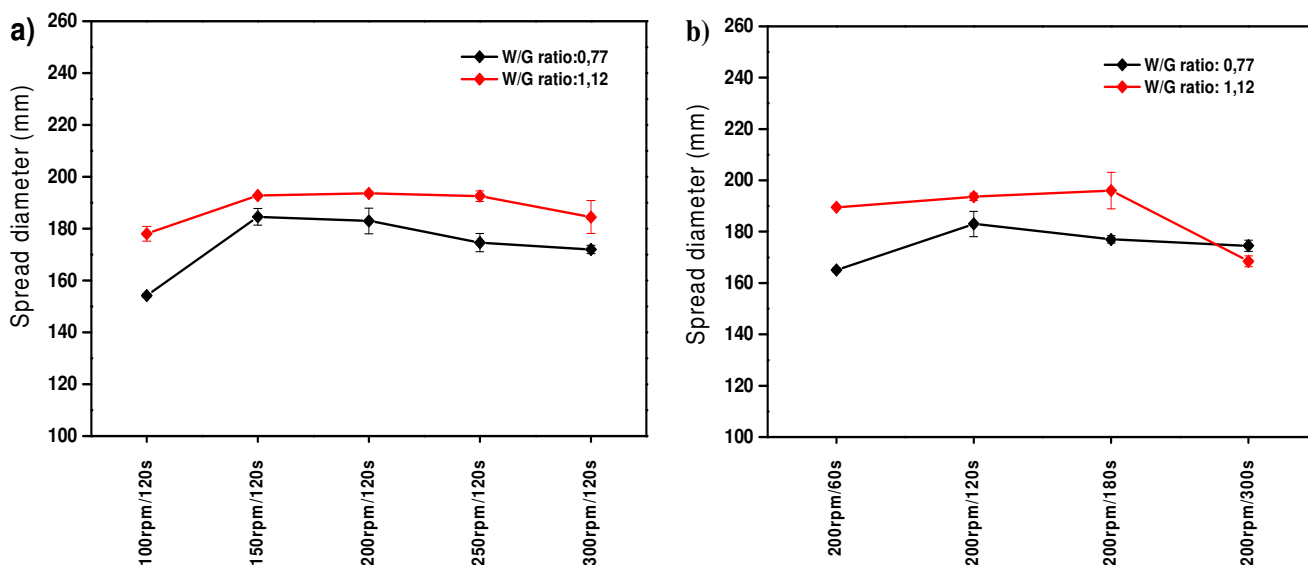


Figure.4. The foams spreadability as a function of a) stirring rate and b) stirring time for different W/G ratios

3.3. Effect of processing conditions on cell morphology

Morphology of mineral foams was observed to examine the effect of stirring rate and stirring time on the foam structure. Figure.5 shows the variation of cell structures at different stirring rates with a constant mixing time of 120 s. The porous structures and cell sizes differed and depended on the selected stirring rate. When the stirring rate increases gradually from 100 rpm to 300 rpm, the porous structure changes from a monodisperse structure with few connections between bubbles (Figure.5A) to a highly connected structure (Figure.5E). In addition, the diameter of the pores increases with the increment of stirring rate. It should be noted that the pore structure (pore size, pore shape, and pore connection) is a key factor that affects foam material properties [6,28].

Additionally, the impact of stirring time on the cell morphology of mineral foam is also investigated (Figure.6). A more uniform interconnected porous structure was obtained with the stirring time from 60 s to 300 s at a constant stirring rate of 200 rpm. On the other hand, increasing stirring time leads to the gradual increase of uniform distribution of pores of smaller diameter between larger pores, conducting to a polydisperse structure. Samples with low density have a high porosity structure. This variation of foam morphology would have an impact on the physical and mechanical properties of the foam.

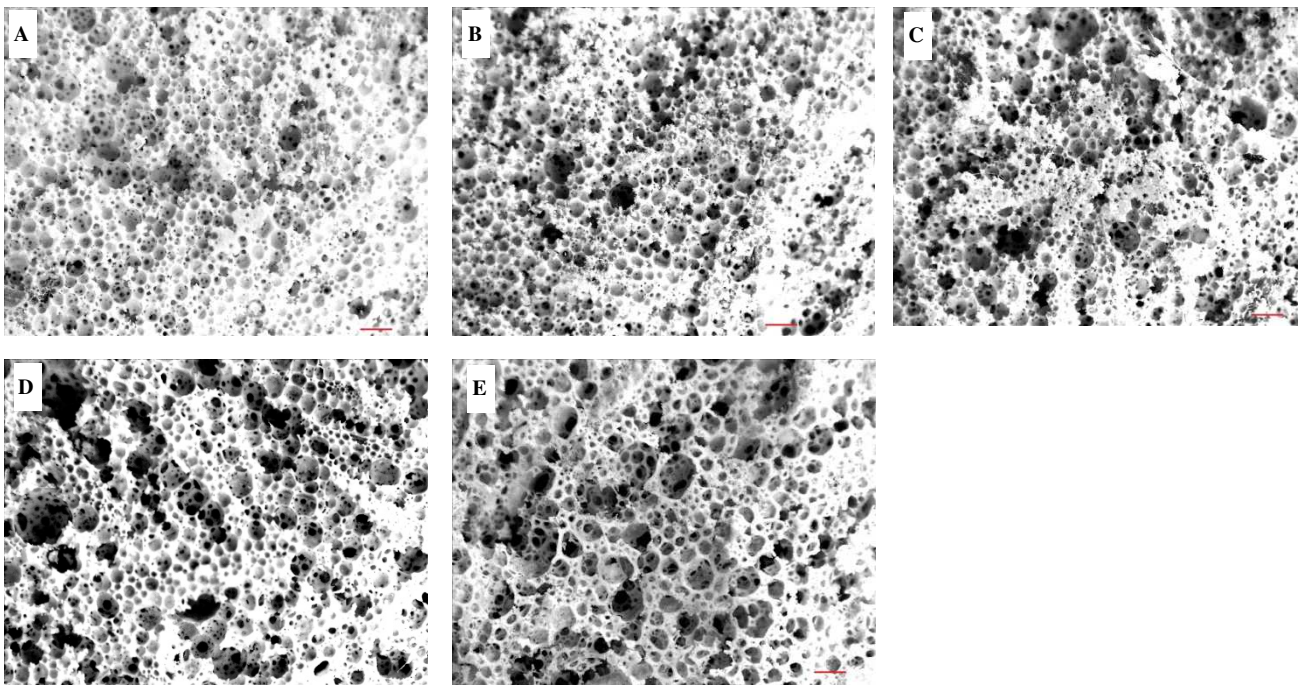


Figure.5. the morphology images of the foams with 1.12 W/B ratio prepared with: (A) 100 rpm/120 s; (B) 150 rpm/120 s; (C) 200 rpm/120 s ; (D) 250 rpm/120 s ; (E) 300 rpm/120 s. the red line in images corresponds to a 1 mm scale.

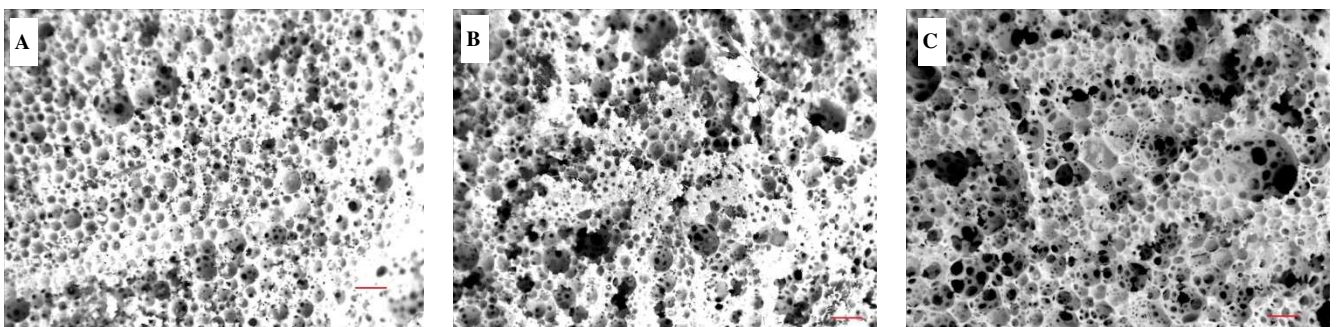
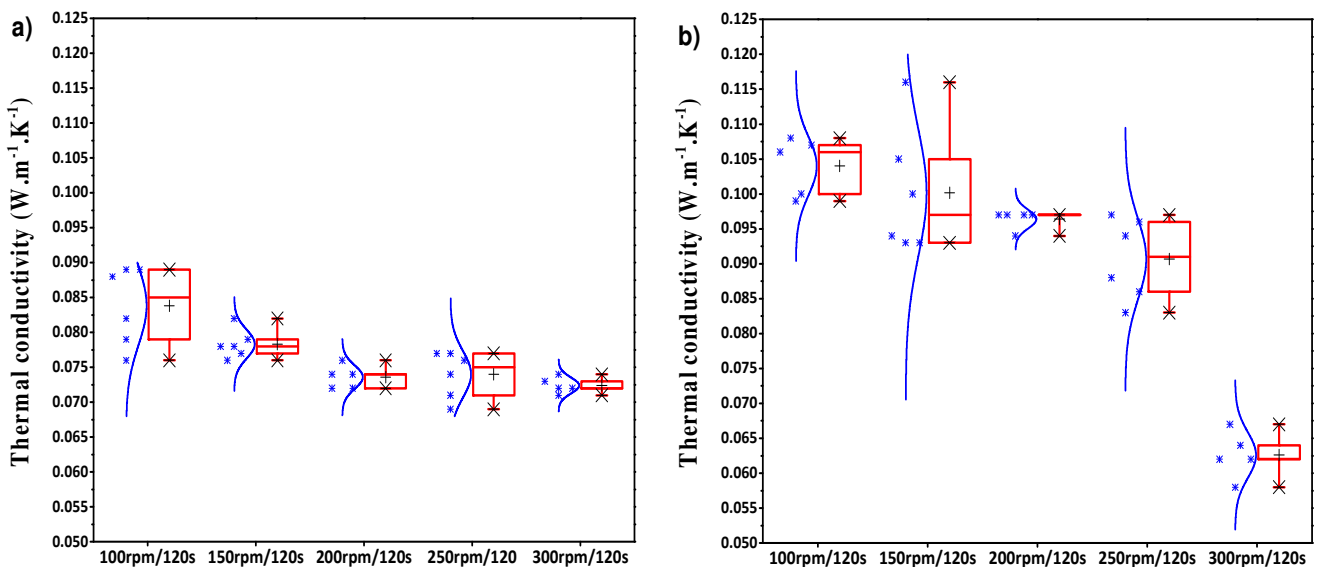


Figure.6. the morphology images of the foams with 1.12 W/B ratio prepared with: (A) 200 rpm/60 s; (B) 200 rpm / 120 s; (C) 200 rpm/180 s. The Red line in images corresponds to a 1 mm scale.

3.4. Effect of mixing conditions on thermal properties

Thermal conductivity λ is an important insulation property of mineral foam. It characterizes the thermal behavior of the foam. Figures.7 present the boxplot results of the thermal performance of different mineral foams prepared with different stirring rates and stirring times. As can be seen in the figures, the variation of stirring rate and stirring time leads to a change in thermal conductivity. Indeed, increasing of stirring rate of the mixer provides foams with low thermal conductivity. For foam prepared with a W/G ratio of 0.77, the thermal conductivity changes significantly ($p < 0.05$) from 0.084 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for 100 rpm to 0.078, 0.074, 0.074 and 0.072 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for 150, 200, 250 and 300 rpm, respectively. As regards foams with W/G ratio of 1.12, the thermal conductivities differ significantly ($p < 0.05$) during mixing rate variation. Thermal conductivities decrease from 0.104, 0.100, 0.096, 0.091 and 0.063 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for 100, 150, 200, 250 and 300 rpm, respectively. This decrease is more stressed when increasing stirring time. In addition, the variation of the W/G ratio strongly affects the thermal conductivity of mineral foam. For example, in the case of foams with a W/G ratio of 0.77, the highest and lowest obtained thermal conductivity are 0.083 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and 0.064 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. In contrast, the thermal conductivities of foams with a W/G ratio of 1.12 are between 0.110 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and 0.063 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. Low W/G ratio results in a reduction of thermal conductivity of mineral foam.



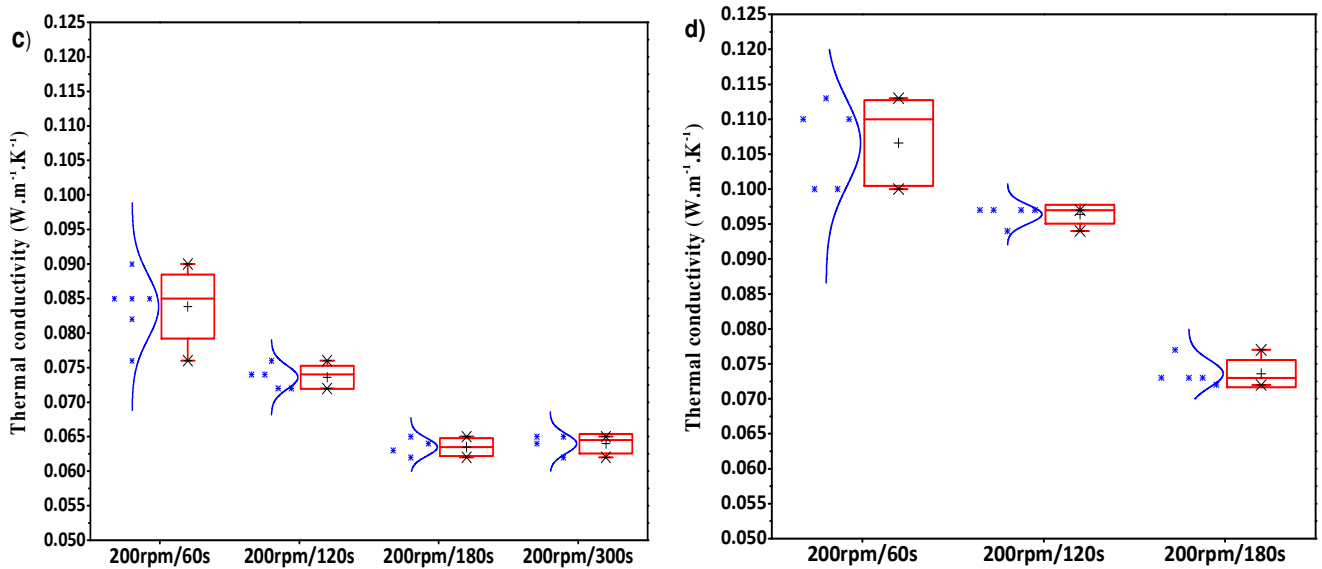


Figure.7. Boxplot of thermal conductivities as a function of stirring rate for foams prepared with (a) 0.77 W/B ratio ; (b) 1.12 W/B ratio. Boxplot of thermal conductivities as a function of stirring time for foams prepared with (c) 0.77 W/B ratio ; (d) 1.12 W/B ratio.

These results highlight that stirring rate, stirring time, and water/binder ratio can affect the material porosity, pore structure, and density, thereby affecting the thermal conductivity value of the material.

The correlation between thermal conductivity and dry density is illustrated in Figure.8. The thermal conductivity varies linearly with the density and followed the equation $\lambda = 2.2 \times 10^{-4}\rho + 0.023$. A decrease in dry density by 100 kg.m^{-3} results in a reduction of $0.045 \text{ W.m}^{-1}.\text{K}^{-1}$ in the thermal conductivity of mineral foam. Other researchers similarly reported that there is a linear relationship between thermal conductivity and the density of lightweight materials [6,19,20]. Low densities with a larger size and wider distributions of voids lead to low thermal insulation capacity [31].

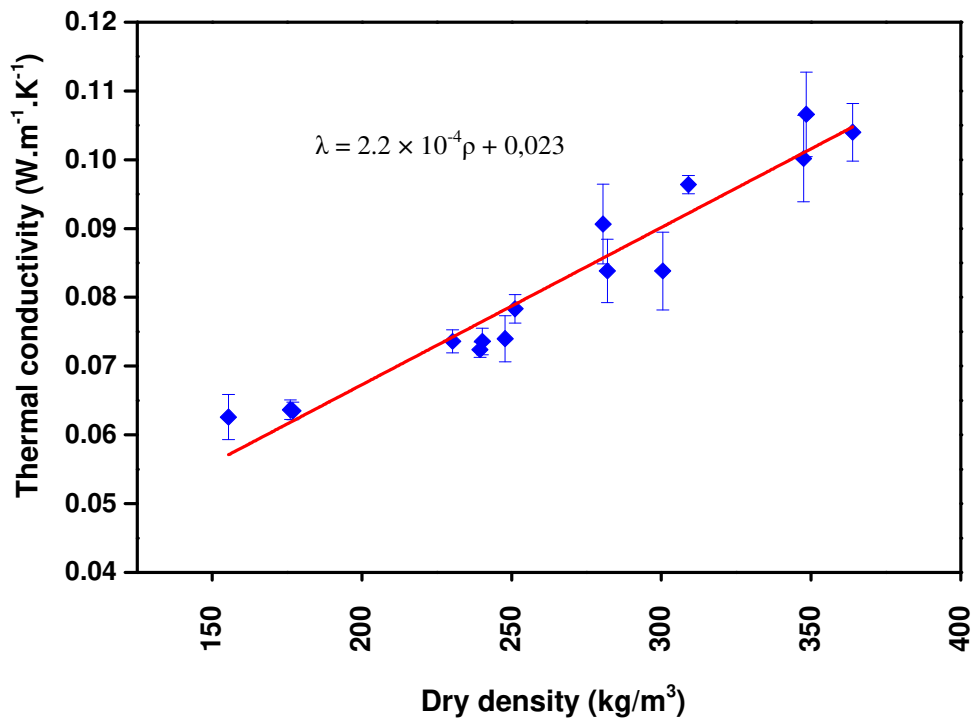


Figure. 8. Mineral foam thermal conductivity as a function of foam density.

3.5. Effect of processing conditions on mechanical properties

Figures.9 and 10 show the effect of mixing conditions on the mechanical properties of mineral foam. As observed in Figures.9a and 9b, mechanical performances of flax shives mineral foams display poor inner strength. The compressive strength values are found to be between 12 to 72 kPa and 15 to 180 kPa for 0.77 and 1.12 W/B ratios, respectively, depending on the stirring rate and stirring time applied during foam manufacturing. In addition, the flexural strength values are between 11-75 kPa for foams prepared with 0.77 W/B ratio and 76-250 kPa for foams prepared 1.12 W/B ratio (Figures.10a and b).

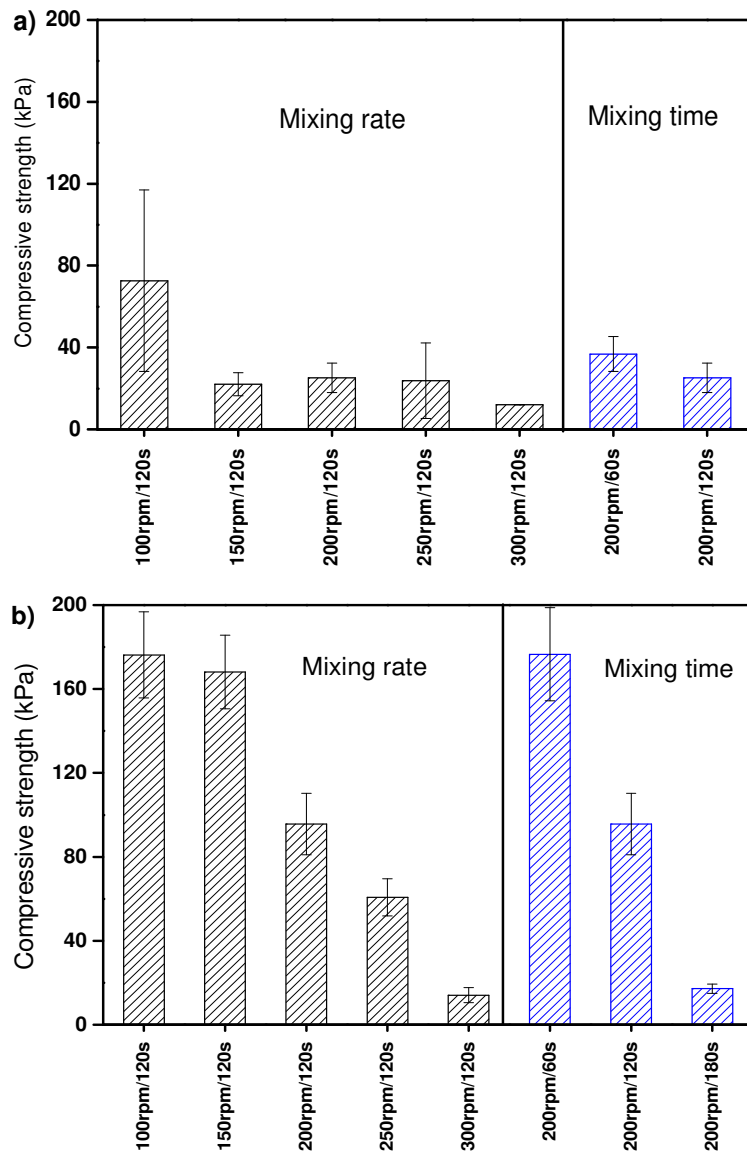


Figure.9. The compressive strength as a function of stirring rate and stirring time for foams prepared with (a) 0.77 W/B ratio ; (b) 1.12 W/B ratio.

Gradual increase of the mixer's stirring rate and stirring time leads to a gradual decrease in compressive strength and flexural strength. It should be noted that the more the stirring rate and stirring time increase, the more the decrease in compressive strength and flexural strength is significant ($p < 0.05$). This decrease of mechanical performances of foam is related to structural changes and reduction of mineral foam density. Increasing stirring rate and stirring time, resulted in less compact mineral foams, with more open pores, which can then impact mechanical performances. Many authors [6,13,14] highlighted that the mechanical performances of mineral foam are generally

affected by the density of materials. The decrease of foam density with the addition of air voids induces a diminution of the strength properties.

Moreover, foams prepared with a W/G ratio of 1.12 present the best mechanical properties compared to foams prepared with a W/G ratio of 0.77 whichever stirring rate and stirring time selected. The samples with a high W/G ratio were more compact and homogeneous than those with low W/G. Therefore, insufficient water amount could be a reason for low material strength. These results are in agreement with that obtained in the literature. Tam et al. [32] and De Rose and Morris [33] reported that the mechanical properties of foams increase at high water-binder ratios.

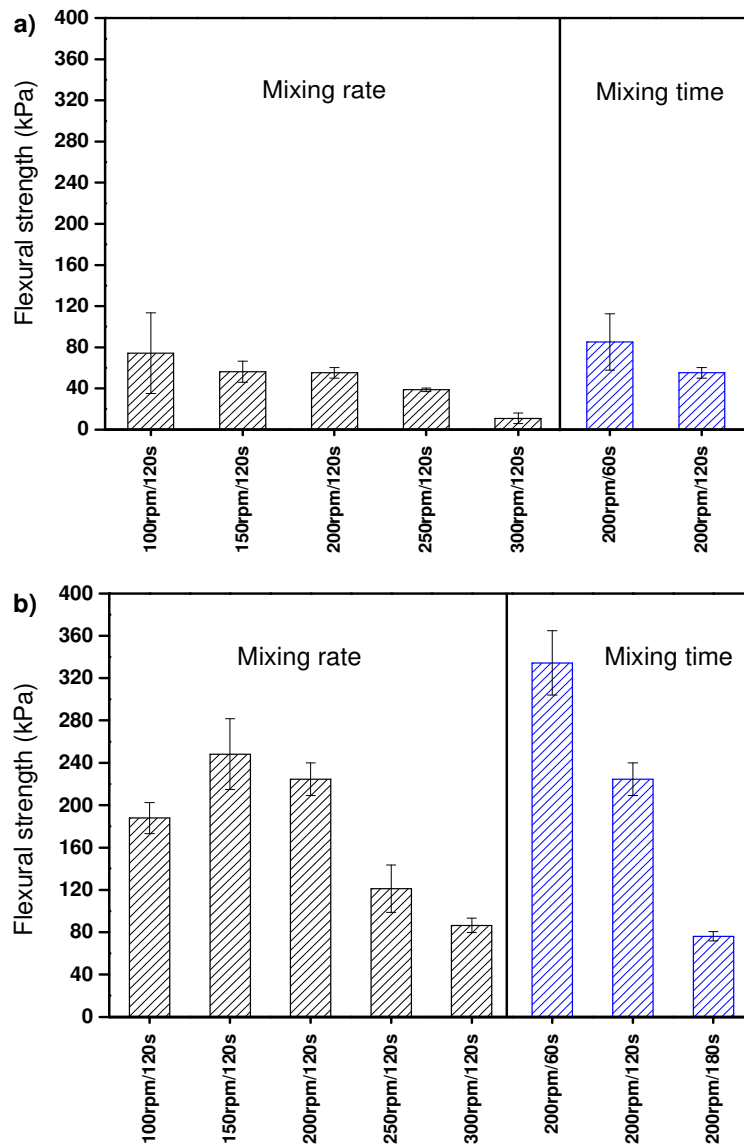


Figure.10. The flexural strength as a function of stirring rate and stirring time for foams prepared with (a) 0.77 W/B ratio; (b) 1.12 W/B ratio.

4. Conclusions

This study investigates the effect of mixing conditions (i.e., stirring rate, stirring time, and W/B ratios) on the density, morphology, thermal and mechanical properties of mineral foam. The results demonstrate that according to the stirring rate, stirring time, and W/B ratio, the performances of mineral foams differ. Varying these parameters will allow for different fresh and dry mineral foam densities: ($244 < \rho < 624 \text{ kg/m}^3$) and ($143 < \rho < 364 \text{ kg/m}^3$), respectively. The increase of stirring rate and stirring time during foam manufacturing led to an increase in the porosity, which resulted in a decrease in the prepared foam densities. This decrease in densities induces i) an improvement in the thermal insulation property of final materials ii) a decrease in mechanical properties (i.e., compressive strength and flexural strength).

In the case of varying W/B ratios, the performances of mineral foams also change. Foams with a high W/B ratio exhibit the best compressive strength and flexural strength values compared to foam with a low W/B ratio. In contrast, low thermal conductivity values were obtained in foam with a low W/B ratio. A compromise between these two properties should be made to select the application of final materials.

Acknowledgments

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