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Original article

Influence of Some Selected Ceramic Fillers on Rheological, Morphological, Mechanical and Thermal Stability Properties of Rigid Polyurethane Foam

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ABSTRACT

This study investigates the influence of limestone, mica, granite, and kaolin on the properties of polyurethane foam. The selected ceramic fillers were pulverized and sieved to obtain particles smaller than 90 µm that were used as reinforcements in the polyurethane matrix in a randomly dispersed mode. The matrix constituents were mixed in the same ratio while fillers were introduced via a one-shot system approach in predetermined proportions of 3–7 wt. %. The work was carried out to identify the optimum filler to be utilized in the production of improved polyurethane rigid foams. The cream, gel, rise, and tack-free times as the rheological properties were observed as the filler materials were introduced in the polyurethane matrix. Mechanical, water absorption and thermal stability properties were evaluated while the morphology of the filled polyurethane foams was examined with SEM. From the results, it was found that kaolin-filled polyurethane foam produced the optimum rheological, mechanical, water absorption, and thermal stability properties. Hence, particulate kaolin of less than 90 µm can be used in the production of green foam for various applications in health, automobile, and household materials.

Keywords: *polyurethane, mineral fillers, rheology, thermal stability, rigid foam, green foam.*

1. Introduction

In recent times, high-performance polymer materials have continuously captured the interest of both researchers and various industries globally [1]. Polyurethane is one of the leading polymers being used in the production of foams for numerous applications in many industries, which makes the demand for polyurethane to be increasing [2, 3]. Polyurethanes are currently one of the fastest-growing polymer technology branches. These plastics have gained prominence due to their extensive use in stiff, semi-rigid, soft, and flexible forms [4]. Polyurethane is a versatile material that is derived from a chemical reaction between isocyanates and polyhydroxyl groups [5]. The formation of rigid polyurethane foams (RPUFs) involves a series of distinct processes: polymerization, expansion, gelling, and blowing. Polyurethane (PU) constitutes a significant portion, approximately 23-29%, of the overall foam production. RPUFs are characterized by their highly cross-linked, three-dimensional polymer structures [6, 7]. These materials are widely used in industries such as the automotive, marine, civil, packaging, and furniture

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industries due to their exceptional mechanical and thermal-insulating properties, low apparent density, and resistance to weather conditions [7, 8]. They are applicable in every sector of human life and activities: offices, transportation, marketplaces, under carpets, household seats, and even in electronics packaging [6, 9, 10]. Their major areas of applications include thermal insulation, acoustic shock and sound absorption, buoyancy, and floating.

Rigid PUR foams offer several benefits, including affordability, minimal environmental impact, low density, and excellent insulating properties. The introduction of the optimal amount and type of filler into the foam structure usually improves the physical and mechanical properties as well as their thermal and acoustic insulating properties while reducing their waste and toxicity to a significant extent [6, 11-12].

Various studies have also shown that the characteristics of rigid polyurethane foam can be greatly improved by the use of various filler materials applied in a predetermined proportion. Dukarska et al. [12] researched on properties of rigid polyurethane foam filled with sawdust from primary wood processing. Based on the findings, it was discovered that the addition of wood flour (WF) up to 10% has a minimal impact on the kinetics of foaming. This allows for a reduction in thermal conductivity, a significant decrease in brittleness, and the maintenance of acceptable dimensional stability. However, such a quantity of WF results in a modest reduction in compressive and bending strengths with an increase in water absorption. It is crucial to note that the obtained results are satisfactory and comparable with the specifications of insulating materials based on rigid PUR foam that are currently in the market, despite the observed drop in the values of these parameters. In another research carried out by Omotoyinbo et al. [6] on comparative investigation of the influence of kaolin and dolomite on the properties of polyurethane foam. Results showed that the density as well as hardness, flexural, and compressive strengths of the polyurethane foam were enhanced with the addition of dolomite and kaolin particles. Effects of kaolin additions on thermal behaviors of rigid polyurethane foams investigated by Aydoğan & Usta, [8] showed that adding kaolin to the foams somewhat enhanced their thermal conductivities and improved thermal stability.

Clay minerals have been extensively used in preparing micronized dispersion on polymer matrix to bring about the desired enhanced properties [13]. However, sodium montmorillonite (Na-MMt) a natural smectite clay (2:1 phyllosilicate) consisting of aluminosilicate layers with a high aspect ratio and a high surface area was subsequently used to synthesize polymer-clay nanocomposites [14, 15]. This research was carried out to investigate the influence of some selected fillers on the properties of polyurethane foam to further identify filler materials that are capable of effectively improving the foam properties. The influence of these fillers on the morphology, mechanical properties, water absorption, and thermal stability of the polyurethane foams was evaluated.

2. Materials and Methods

2.1 Starting Materials and Processing of Foams

The basic polymeric materials (isocyanate and polyol) utilized were procured were manufactured by Dow Chemicals in the United States. Mica, limestone, granite, and kaolin particles were chosen as ceramic filler materials. A laboratory ball mill was used for grinding the ceramic materials to powder form and sieved to obtain particles smaller than 90 μm that was used.

The polyurethane foam was developed by mixing the two components which have been pre-blended. Both components (isocyanate and polyol) were mixed in a ratio of 1:1 with 60 g of each weighed in a plastic cup. The non-filled foam was first developed to serve as the control sample. For the composite samples, fillers (particles smaller than 90) were added to polyol and manually mixed properly for 5 minutes before adding isocyanate and mixed for another 5 minutes. At this time, the mixture was observed to have turned creamy. The creaming time was estimated with the aid of a digital stopwatch. Following this was the gelling of the paste, at this point the mixture was quickly poured into the mold, and the homogenous paste started to rise. The gel time was also determined and recorded. The cast was allowed to rise to its maximum height unhindered till it was tack-free and was de-molded after 30 minutes. This process was carried out in accordance with Omotoyinbo et al. [6]. The samples were then cut into the appropriate shapes and sizes for property assessment. The fillers were added in three proportions (3, 5, and 7 wt. %). The time it took each sample to reach its cream, gel, rise, and tack-free stage was recorded.

2.2 Characterization of processed foam

2.2.1 Structural Constituent

Microstructural analysis of the unfilled and mica, kaolin, granite, and limestone-filled polyurethane foams was carried out using a field emission scanning electronic microscope (JEOL JSM-760 0F). An FT-IR spectrometer (Infrared spectrometer Varian 660 MidIR Dual MCT/DTGS Bundle with ATR) was used to confirm the chemical structure of all samples. Before analysis, the samples were dried in an auto-desiccator for 24 hours. Samples were directly applied to a diamante crystal of ATR and the resulting spectra were corrected for background air absorbance. Potassium bromide (KBr) disks were prepared from powdered samples mixed with dry KBr in the ratio of 1:100. The spectra were recorded in a transmittance mode from 4000 to 500/400 cm^{-1} at a resolution of 4cm. The infrared spectrum was Fourier transformed and recorded in the absorption mode.

2.2.2 Characteristics time

A cup test was used to determine the characteristic times, such as cream, gel, rise, and tack-free times of the polyurethane foam production.

2.2.3 Water absorption test

A water absorption test was carried out on all sectioned samples of the filled foam and the control samples. The samples were immersed in water for 4 days where the samples were re-weighed every 24 hrs. and their masses recorded. The initial weights (w1) were measured and recorded before the immersion and the final weight of the foam (w2) was used in deducing the moisture content. The moisture content of the foams was calculated using Equation (1) as reported in research carried out by Vargas-Gutiérrez et al. [16]:

$$\text{Water absorption (\%)} = \frac{w2-w1}{w1} * 100 \quad (1)$$

2.2.4 Thermal stability

The thermal stability for each of the foam was calculated by sectioning a representative of the whole sample. The samples were placed into the laboratory oven and subjected to a temperature of 70˚C for 30 minutes and afterward volumetric, density, and weight changes.

2.2.5 Hardness test

The hardness of the foam samples was measured by a model LX-DYJ a Shore D hardness measuring equipment in accordance with ASTM D2240-00 having its needle stroke equaling 2.5 mm by length, needle head size: SRO 1 mm. Its gauge conforms with GB/T531, GB/2411, HG/T2489, and JB/6148 regulations. The foam samples' hardness was measured at different spots, with an average value representing the individual foam hardness value.

2.2.6 Compression test

A compression test was carried out on the samples after cutting them into cubes with dimensions of 30 cm x 30 cm x 30 cm. They were tested using an automated universal testing machine of model 3369 INSTRON running on Blue Hill Two software. The raw data were obtained, and the strain-strain relationship was established using the finite difference method as reported in Omotoyinbo et al. [6].

2.2.7 XRF Analysis

X-ray fluorescence (XRF) analysis was carried out on the ceramic fillers to determine their elemental compositions using an Energy Dispersive X-ray Fluorescence spectrometer (Skyray Instruments, EDX3600B). The chemical analysis was performed using about 0.5 g of the sample material pressed using lithium tetraborate (flux) which allows evenly dispersed solid solution. The x-rays beamed on the sample and caused excitement on some of the atomic nuclei to jump to the higher energy orbitals to facilitate the identification of the elements in the sample.

2.2.8 Microstructural analysis

Microstructural analyses of the unfilled and polyurethane foams were carried out using a field emission scanning electronic microscope (JEOL JSM-760 0F). The accelerating voltage of the microscope is 15 kV. Before the analysis, the samples were gold-sputtered to improve electrical conductivity.

3. Results and Discussion

3.1 Cream time

In the production of rigid foam, the cream stage is the first reaction stage between the reactants (polyol, isocyanate, and filler). The cream time is the time for the reactants to achieve a homogenous mix and produce a cream-like color. The cream time of the samples varies and mainly depends on the quantity of the matrix components used as well as the wt. % of filler.

From Fig. 1, it was observed that the cream time of the fill samples increases compared to that of the unfilled sample (control). Also, it was discovered that all the filler materials except for granite influence the cream time in a similar mode. The fillers have their peak values at 5 wt.% while granite had its least value at 5 wt% to emerge as the minimum cream time for the filled polyurethane with a value of 5.71 s next to that of the control with a value of 3 s. The highest cream time of 18.7 s was attained from kaolin-based polyurethane foam. Hence, from the results, it was revealed that the presence of fillers tends to increase the number of seconds it takes for the reaction to reach cream time compared to pure polyurethane foam. This implies that there is a delay in the chemistry of the polyurethane for samples containing fillers than that of the sample containing no filler and this is because fillers often act as nucleation sites, promoting controlled expansion and influencing the foam's viscosity.

3.2 Gel time

This is characterized as the period when bubbles are formed or when the reaction starts developing or rising. The samples achieve a gel state after the homogenous mix forms into a pasty substance. The time taken for samples to gel directly affects their rise time. From Fig. 2, it was observed that the addition of fillers to the mixture led to an increase in the gel time compared to the control sample, resulting in a delay. It was also observed that the control sample had the least gel time of 14.03 *s* compared to the filled polyurethane foams similar to what was observed in Fig. 1. This implies that it took the control sample a total transition time of 11.03 s to achieve a gel state having the shortest delay compared to the filled samples. It means that the control sample achieved its gel state faster than the filled sample. Among the fillers, limestone and granite have similar effects on the foam while mica and kaolin have different effects. Again, granite with 5 wt.% gave the least gel time of 23.29 s among the filled polyurethane while kaolin with 5 wt.% gave the maximum gel time of 38.22 s compared to the control sample. The reasons for these are also similar to what was stated in Fig. 1.

3.3 Rise time

After the gelling of the mixture, which forms a paste, it rises due to continuous reactions, which lead to rigid polyurethane foam. The influence of some of these fillers in weight percent brings about a faster rise time. This is highly significant for improving the foam curing time.

From Fig. 3, it was observed that mica and granite have similar effects on the filled polyurethane foams while others have varying effects with samples from 3 wt. % kaolin having the least rise time of 49.34 s compared to the control sample with a value of 51.86 s. This could mean that the sample had the fastest rising time because the kaolin may have facilitated efficient nucleation and cell growth during the foaming process and this could lead to quicker expansion and solidification, resulting in the lowest rise time. The rapid foaming process is a desirable property of foam during production and, thus, kaolin can be considered for such input in foam making. However, a sample from 7 wt. % mica had a peak value of 69.88 s compared to the control sample. It was also observed that all samples from mica (3–7 wt. %) had longer rise times than the control sample. This indicates that mica samples exhibited a lower curing rate than the control sample and may have contributed to increased viscosity and hindered the flow during the foaming process. This might have led to a slower rise and expansion, resulting in the highest rise time, which may not be desirable.

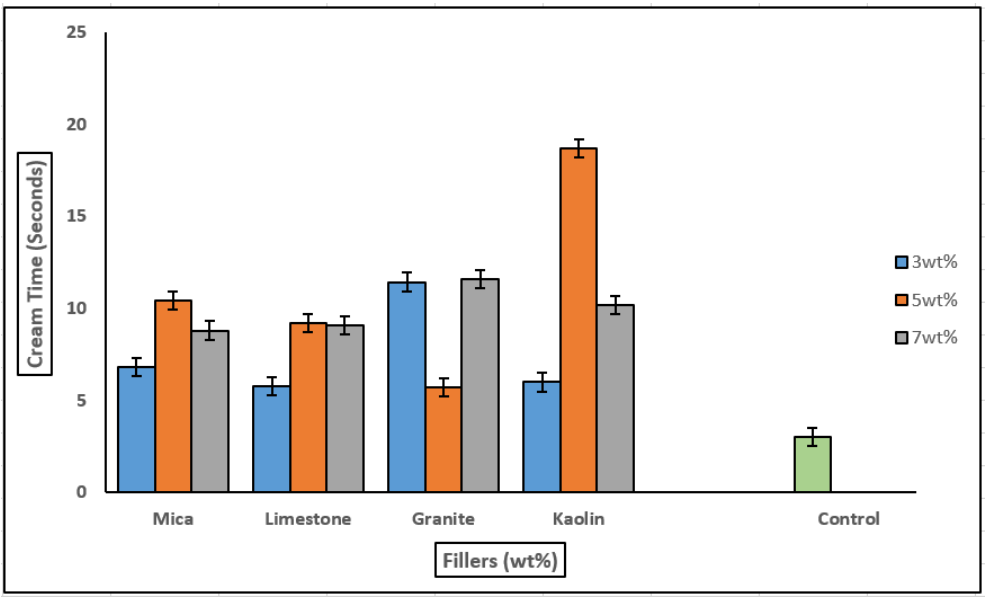


Fig. 1 *Cream time of the filled & unfilled polyurethane foam*

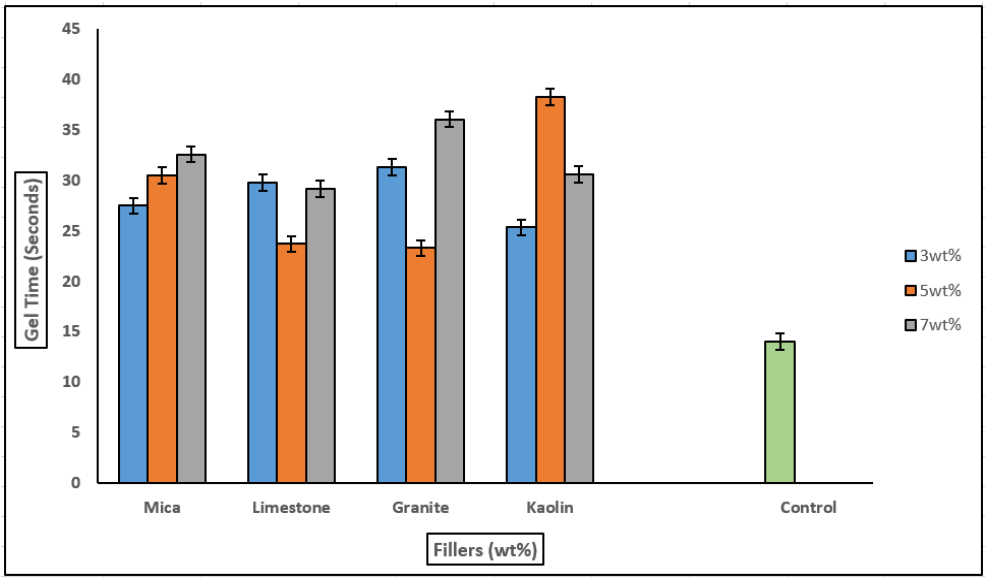


Fig. 2 *Gel time of the filled & unfilled polyurethane foam*

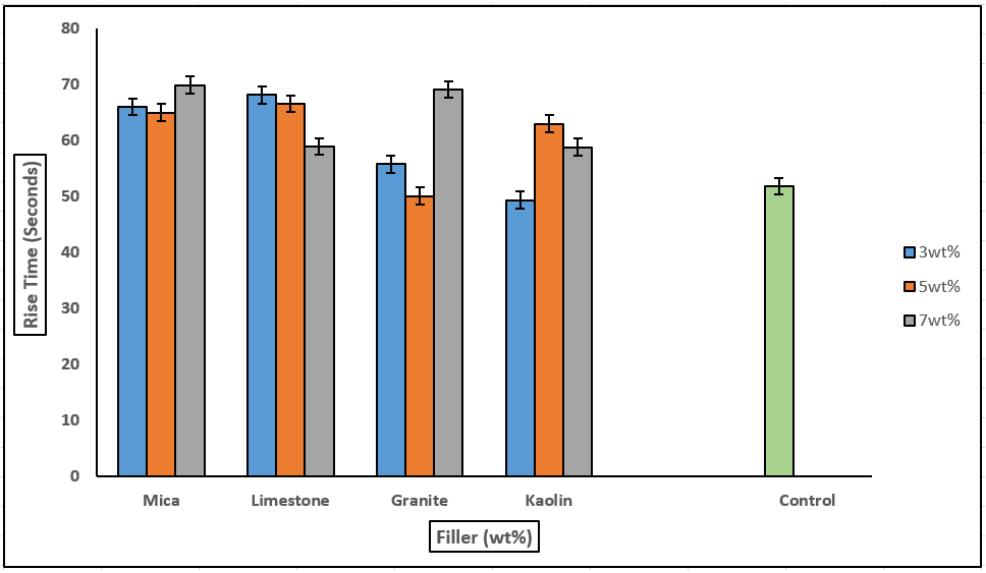


Fig. 3 *Rise time of the filled & unfilled polyurethane foam*

3.4 Tack free time

As the foam is formed, it possesses a sticky nature, which gradually fades away as the foam cures. This sticky property can be observed by touching the foam surface. The rise time of the various samples directly affects the time taken for the sample to become completely tack-free, which signifies that the faster the rise time, the faster the sample becomes tack-free, which in turn results in a shorter curing time for the foam.

Similar trends to Fig. 3 were observed in the results from Fig. 4, where it was observed that, a sample with 3 wt. % kaolin had the lowest tack-free time of 58.87 s compared to the remaining samples, including the control sample with a value of 62.89 s. This means that the sample surface can be touched without sticking, and all the physical properties are completely established within the foam. Hence, it can be stated that kaolin, being a clay mineral, promotes efficient curing and solidification, resulting in a faster transition to the tack-free state. Samples from mica (3-7 wt.%) gave the highest tack-free times when compared to others but the peak value was obtained from the sample with 3 wt.% from limestone with 83.42 s. The reasons for the observed behaviors are similar to what was stated in Fig. 3.

Considering the rheological properties of the developed polyurethane-based composites within the range of the filler contents, it was discovered that cream and gel times followed the same trends with the filler additions while the rise and tack-free times followed the same trends with respect to the fillers. It was revealed that 5 wt.% kaolin-based was the sample with optimum cream and gel times while 3 wt.% kaolin-based polyurethane foam was the sample with the minimum rise and tack-free times to give the best desirable results.

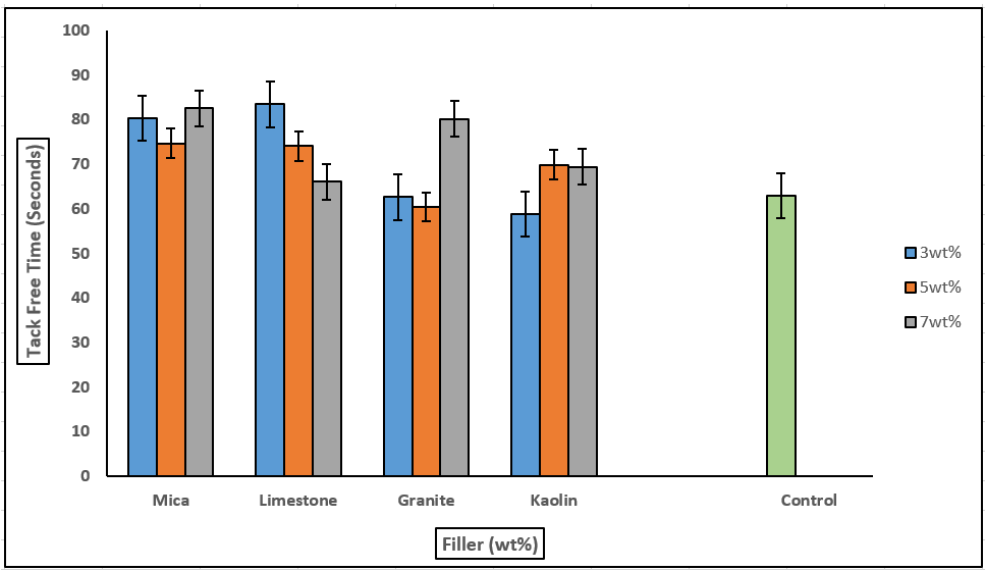
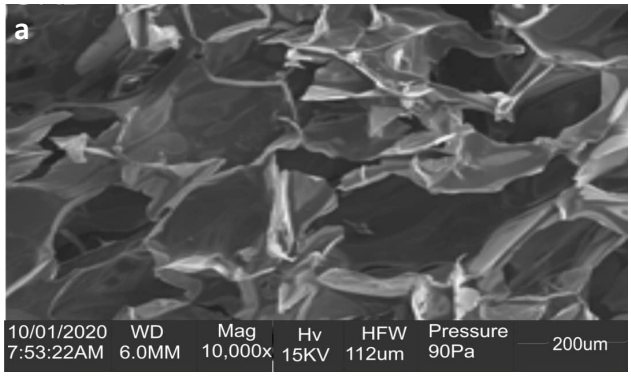
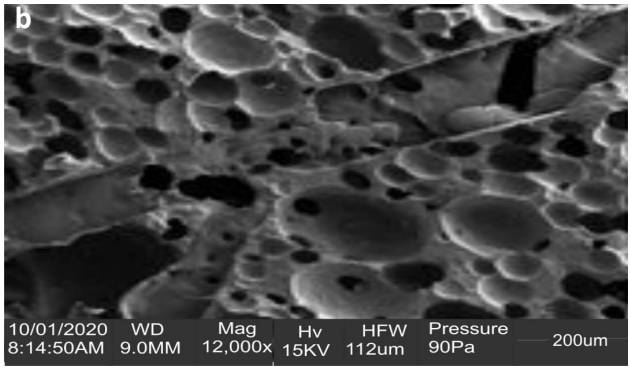


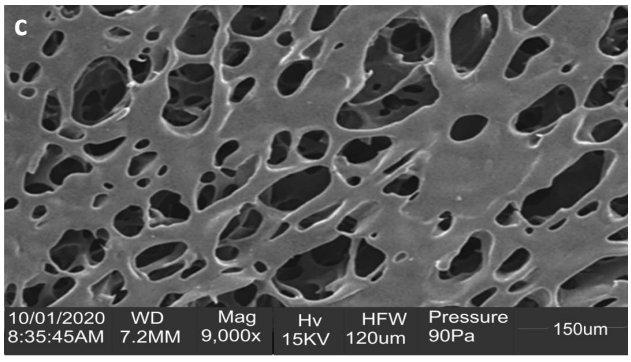
Fig. 4 Tack free time of the filled & unfilled polyurethane foam

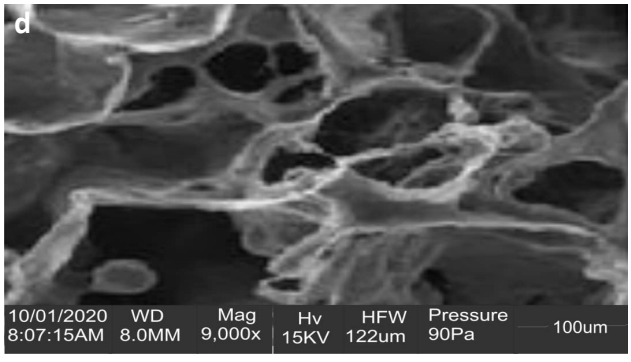
3.5 Microstructural characterization of filled and unfilled polyurethane foam

Figure 5(a-e) shows the SEM micrographs of the unfilled polyurethane, granite, limestone, kaolin, and mica-filled forms.









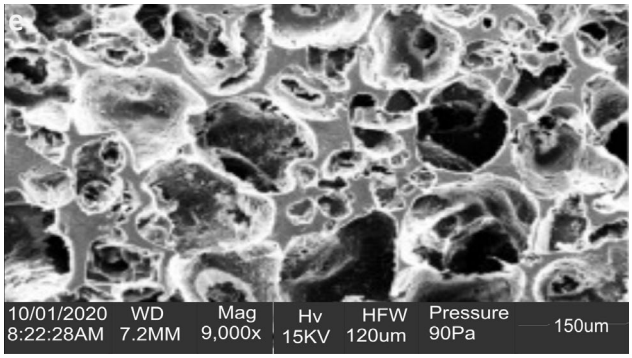


Fig. 5 *SEM micrographs of samples (a) unfilled polyurethane foam (b) granite-filled polyurethane foam (c) limestone filled (d) kaolin filled (e) mica-filled polyurethane foam*

The Figure revealed the microstructural images of the features due to the interactions between the polyurethane matrix and the filler surfaces. It was clear from Figure 5(a) that, pure PUR foam has a structure with a significant proportion of closed cells that has a grain size of 200 µm with a uniform cell structure similar to the finding in previous research as seen in Omotoyinbo et al. [6], Dukarska et al. [12], and Tiuc et al [17]. The addition of fillers into the matrix as shown in Figure 5(b-e) causes the cell structure to be no longer uniform and these fillers added may act as nucleation sites in agreement with Uram et al. [5]. The average grain size for each of the fillers used was between 100-200 µm, where limestone, kaolin, and mica-filled forms had smaller grain sizes when compared to the unfilled polyurethane foam and it was observed that the morphology for limestone-filled foam is quite different from the other microstructures. The differences in morphological features support the reasons for the variations in the observed properties of the prepared ceramics filler-reinforced polyurethane rigid foam samples.

3.6 XRF analysis of filled polyurethane foam

The results of the XRF for the fillers used are shown in Fig. 6(a-d). According to the results, the main element in the granite-filled polyurethane foam is silicon, with a content of about 20% as shown in Fig. 6 (a). Other elements such as iron (about 9%), potassium (about 7%), calcium (about 3%), and aluminum (about 4%) were also present in high proportions. Also, as indicated in Fig. 6 (b), the elements that dominate the mica-filled polyurethane foam include potassium (13%) and silicon (12%). Other elements, such as aluminum (8%) and iron (7%), were detected in higher concentrations than trace elements. According to Fig. 6 (c), calcium (69%) predominated the limestone-filled polyurethane foam. Other elements such as sulfur (2%), aluminum, and silicon (1%) were also present. Fig. 6 (d) shows silicon (25%) and aluminum (23%) predominated the kaolin-filled polyurethane. Elements such as sulfur and calcium (3%) were also present in high proportions compared to trace elements. Material behavior will be influenced by these elements.

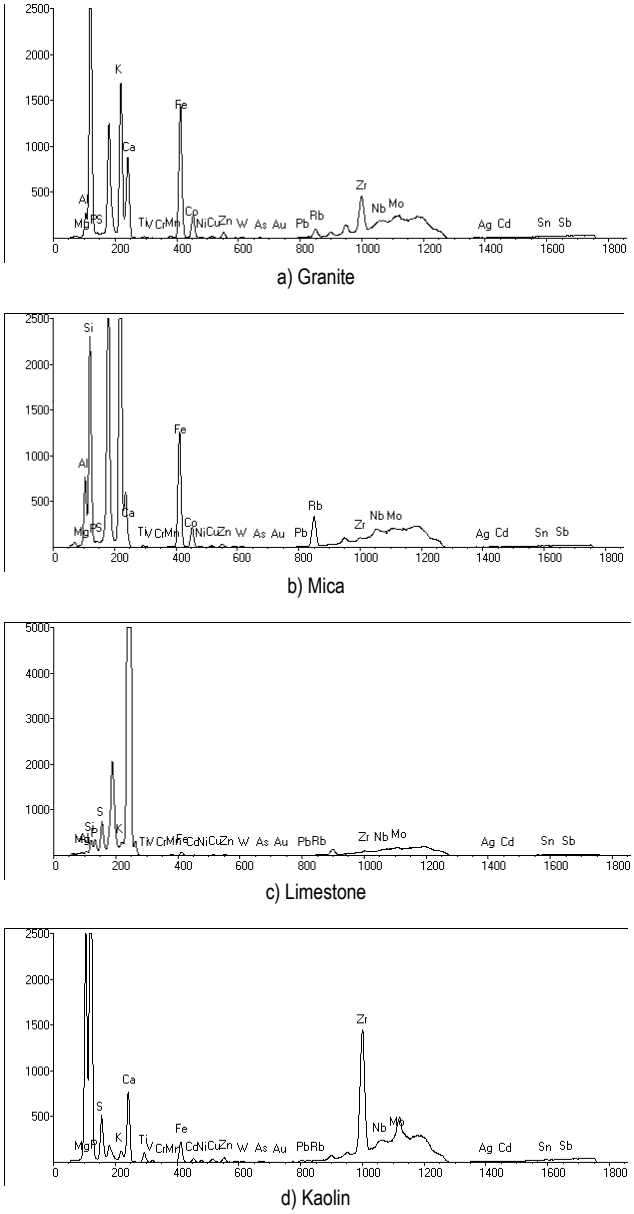
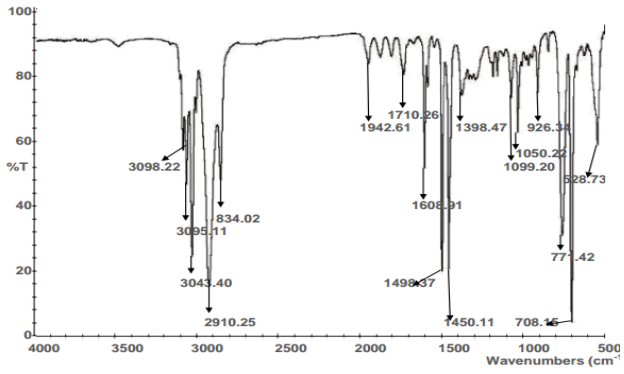


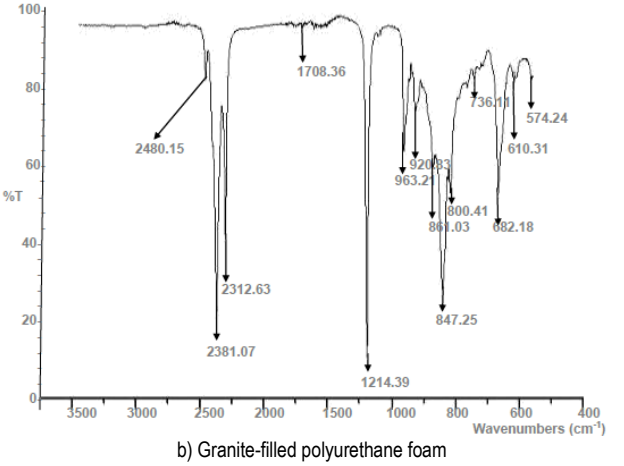
Fig. 6(a-d) XRF Peaks for the selected ceramics fillers

3.7 Fourier Transform Infrared Spectrophotometer (FTIR) analysis

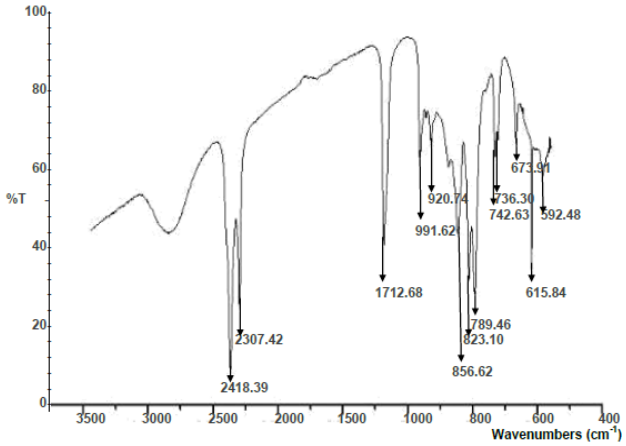
FT-IR measurements were used to investigate changes in composition and chemical functional groups in filled polyurethane foam. The FT-IR spectra of (a) unfilled polyurethane foam, (b) polyurethane foam filled with granite particles, (c) polyurethane foam filled with mica particles, (d) polyurethane foam filled with limestone, and (e) polyurethane foam filled with kaolin particles are shown in Fig. 7. The polyurethane foam in Fig.7a without fillers exhibited two O-H groups responsible for stretching vibration and stretching at 3098.22 and 2910.25 cm^{-1}, respectively. The foam also exhibits a distinct C-O-C peak at 1099.20 cm^{-1}, with chain extenders composed of sulfonic groups. S-H, S=O, and O-S-O peaks emerge at 1710.26, 1398.47, and 1050.22 cm^{-1}, respectively, with the O-S-O peak partially overlapping the C-O-C band. The urethane carbonyl peaks and the H-NH band occurred with varying intensities. At 926.34cm^{-1}, -CH bending vibrations caused by substitution groups in alkenes and aromatics were also seen. The presence of multiple peaks in the unfilled foam indicates that both functional and sulfonic acid groups were successfully incorporated into the polyurethane backbone. The addition of graphite, mica, limestone, and kaolin, as shown in Fig. 7(b-e) causes a change in the modification of the absorption peaks. The O-H group, which is responsible for the stretching vibration, was found to be absent in all of the filled foam except kaolin-filled foam, which has a peak of 2420.38cm^{-1}, while the O-H stretching was found to be present in all of the filled foam except kaolin filled foam, which had a peak of . 2418.39-3086.74cm^{-1}. Aside from kaolin-packed foam, which was also absent, the foam shows a definite C-O-C peak between 800.03-963.21cm^{-1}. This could be owing to the high silicon content. The sulfonic acid groups found in empty polyurethane foam were not found in any of the filled polyurethane foam. The high presence of silicon in granite and kaolin, calcium in limestone, and potassium and silicon in mica, which fostered the formation of the Si-O stretching vibration band, may have contributed to the degradation of the sulfonic acid groups. A similar result was observed in Omotoyinbo et al. [6].



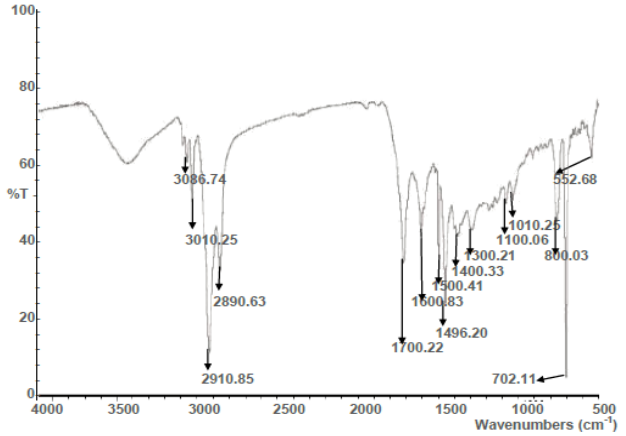
a) unfilled polyurethane foam



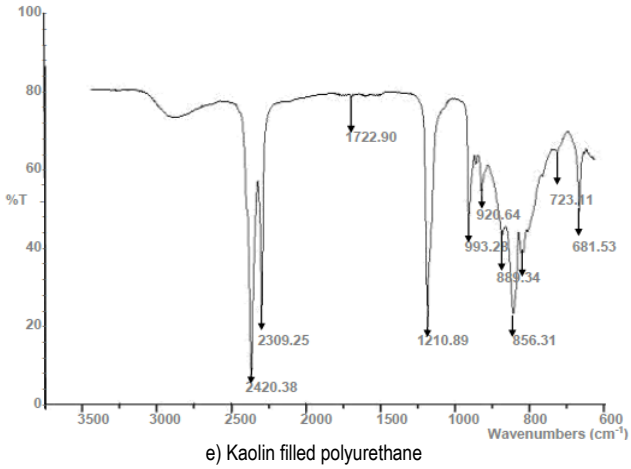
b) Granite-filled polyurethane foam



c) Mica-filled polyurethane foam.



d) Limestone filled polyurethane foam.



e) Kaolin filled polyurethane

Fig. 7(a-e) *FTIR spectra for unfilled and filled polyurethane foams.*

3.8 Hardness property

The hardness properties of limestone, mica, granite, and kaolin-based polyurethane were comparatively investigated, as shown in Fig. 8. Notably, it was observed that the hardness of polyurethane foam, comprising specific weight percentages of fillers (3 wt% kaolin, 3-7 wt% limestone, 3-7 wt% mica, and 5-7 wt% granite), were lower compared to pure polyurethane foam, registering a hardness value of 2 HS. This disparity in hardness can be attributed to several factors. Firstly, fillers such as limestone and mica exhibit a non-reinforcing nature, especially at lower concentrations, where their marginal increase in foam density may not significantly impact hardness. Moreover, their presence at lower concentrations could interfere with the formation of cross-links or bubble structures within the foam, leading to reduced stiffness and hardness. Additionally, poor adhesion between filler particles and polymer chains at lower concentrations may weaken stress transfer mechanisms, further contributing to decreased hardness. Similarly, the reduction in hardness observed with 5-7% granite can be ascribed to factors such as saturation of reinforcing effects, interference with cross-linking, decreased polymer content, and limited compatibility between granite and the polymer matrix. Conversely, polyurethane foam incorporating granite at 3-5 wt% and kaolin at 5-7 wt% exhibited enhanced hardness compared to pure polyurethane foam, with 7 wt% kaolin yielding the optimum hardness value of 2.63 HS, representing a 31.5% increase, relatively. This improvement can be attributed to the addition of these fillers at adequate concentrations, particularly 7 wt% kaolin, which enhances the overall density of the foam, thereby contributing to higher hardness. This may be based on the establishment of a more robust and structurally stable network. Furthermore, an upward trend in hardness across the filled foams was observed with increasing weight percentage of fillers from kaolin and limestone, attributable to their role as internal reinforcements within the polymer matrix. These fillers form physical connections with the polymer chains, resulting in higher stiffness and hardness. The reinforcing effect was more pronounced with increasing filler content, up to a certain threshold, beyond which interference with cross-linking may occur.

However, for foams containing mica as a filler, it was noted that as the weight percentage increased from 3–7 wt%, the hardness decreased. This phenomenon can be attributed to the flat, plate-like morphology of mica particles, which may not effectively interlock or form strong connections with the polymer chains compared to fillers like kaolin or limestone. Consequently, their reinforcing effect is limited, particularly at higher concentrations. Also, mica's hydrophilic surfaces render them less compatible with the hydrophobic polyurethane matrix, weakening interfacial bonding and hindering effective stress transfer, ultimately resulting in reduced stiffness and hardness. This observation aligns with similar findings reported by Omotoyinbo et al. [6] concerning the addition of dolomite and kaolin fillers to polyurethane foam at different proportions (3, 5, and 7 wt%). Therefore, for applications necessitating high hardness, such as orthopedic foams, kaolin-based foam emerges as a viable option.

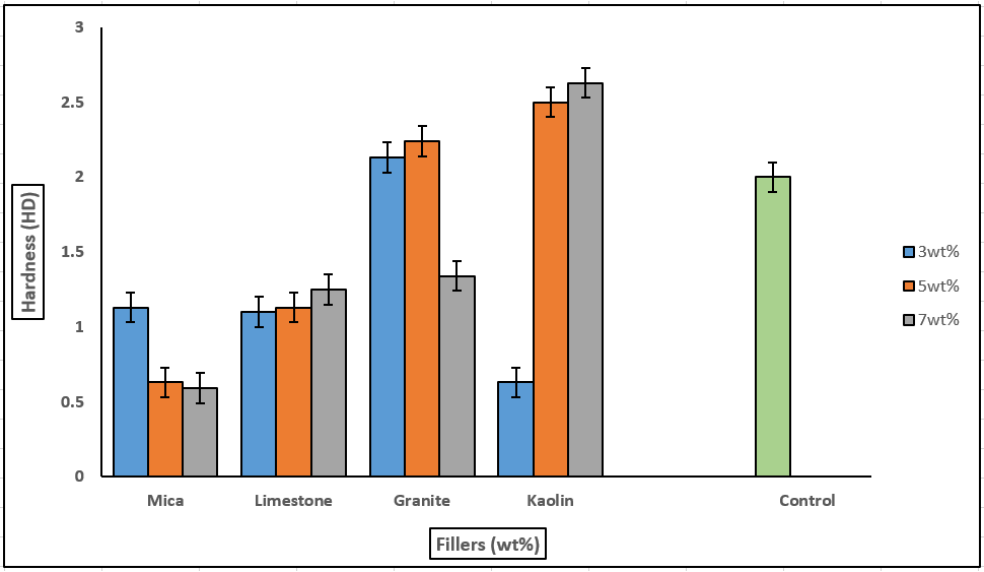


Fig. 8 *Hardness property of the filled & unfilled polyurethane foam.*

3.9 Compressive strength of the polyurethane foam

Fig. 9 illustrates the maximum compressive strength values for both foam samples with and without fillers. It was observed that the compressive strength decreased with increasing weight percentage for fillers such as limestone, mica, and granite, except for kaolin filler. This decrease in compressive strength can be attributed to some of the reasons highlighted in the discussion of Figure 8 based on the addition of these fillers to the polyurethane which leads to weak spots within the foam structure and reduces its ability to withstand compressive loads. In addition, the presence of mica filler at higher concentrations may affect the nucleation and expansion dynamics of bubbles, resulting in larger air cells. This ultimately reduces the compression. Furthermore, the hydroxyl groups on the surface of kaolin can interact with the isocyanate component of the polyurethane during the cross-linking process. This interaction promotes the formation of a denser and more interconnected polymer network, resulting in higher compressive strength. Moreover, it was found that foam samples with specific weight percentages of fillers (limestone 7 wt%, mica 5-7 wt%, granite 7 wt%, and kaolin 3 wt%) exhibited lower compressive strength compared to pure polyurethane foam (0.129 MPa). This decrease can be attributed to similar factors, as discussed in Figure 8. However, the remaining foam samples with different weight percentages of fillers showed higher compressive strength, with the 7 wt% filler content demonstrating the optimum compressive strength of 0.167 MPa, representing a 29% increase. density of the foam and creates internal voids which can have a negative impact on the compressive strength. On the other hand, the increase in compressive strength observed for kaolin filler can be attributed to its natural plate-like structure, which effectively interlocks with the polymer chains of the polyurethane matrix. This interlocking creates a more robust and interconnected network, enhancing the material's resistance to deformation under This improvement can be attributed to the balanced reinforcement provided by the 7 wt% filler content, which increases the density of the foam without introducing a significant number of large air cells that would weaken the structure. Similar findings were also reported by Omotoyinbo et al. [6].

3.10 Water Absorptivity

Fig.10 illustrates the moisture content in terms of water absorption potentials for both unfilled and filled foam. The moisture contents after 24, 48, and 72 hours are represented by M1, M2, and M3, respectively which is similar to that of Dukarska et al [12]. According to Dukarska et al. [12], rigid PU foams exhibit hydrophobic properties and possess closed cells. Consequently, water infiltration is limited to the pores found between foam cells[18]. As depicted in Figure 10, the introduction of fillers into the foam results in a gradual increase in absorption. This phenomenon can be attributed to disturbances in the foam's morphology, specifically an increase in the number of open cells that have a greater capacity to store water, which aligns with the findings of Strakowska et al. [19] and Oladele et al. [20]. Notably, after 24 hours, the moisture content of the filled foam surpasses that of the unfilled foam, except for foams based on limestone and granite. Consequently, it was disclosed that the limestone-based foam with a weight percentage of 7% exhibits the lowest water absorption value of 226% after 72 hours, in contrast to the unfilled polyurethane foam with a value of 268%. This disparity can be attributed to the higher calcium content present in the limestone-based foam, as well as its distinct closed morphological feature, as observed in Figure 5. However, the sample consisting of 5% mica-filled foam demonstrates the highest moisture content after 72 hours, amounting to

approximately 334%. Consequently, the foam containing 5% mica exhibits the optimum water absorption rate, whereas the foam filled with 7% limestone displays the lowest water absorption rate.

3.11 Thermal conductivity

Thermal stability for both filled and unfilled foam was presented in Fig.11 where M1 represents initial mass and M2 represents final mass after being raised to a temperature of 70°C for 30 minutes. Compared with the unfilled foam sample, there were no significant differences in the mass of the foam and it was discovered that foam with 3 wt.% mica-filled had a stable mass of 1.45 g similar to the control that had 1.11. The high thermal stability of mica could be due to the presence of high potassium content compared to other fillers. However, the remaining filled foam samples have slight differences between the initial and final masses which fall within 0.01-0.02. As reported by Borkowski [21], polyurethane materials with closed-cell structures should not change linear dimensions by more than 1.5% and geometric volume by more than 3%. These requirements indicate a high level of stability for filled foams which these filled foams have met, and, thus, are thermally stable.

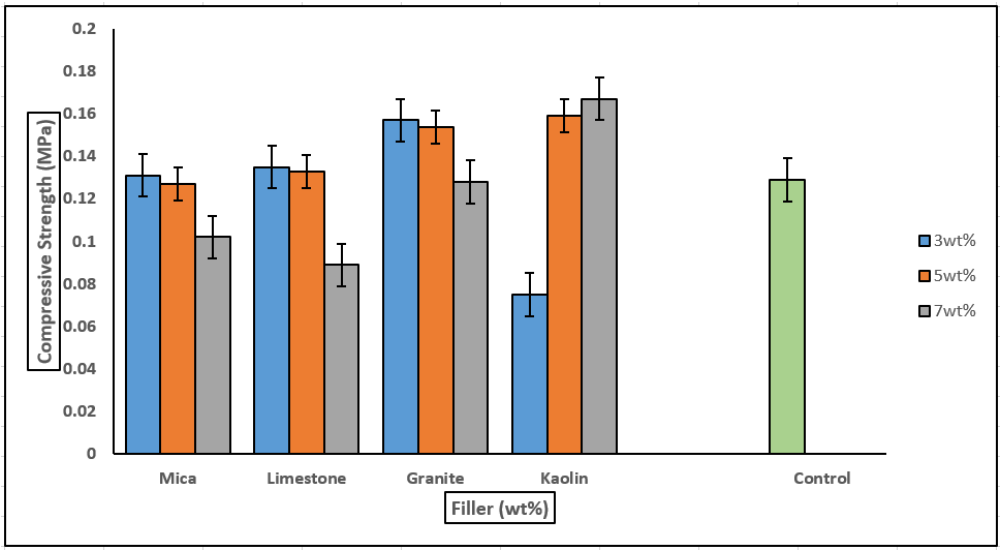


Fig. 9 *Compressive strength of the filled & unfilled polyurethane foam.*

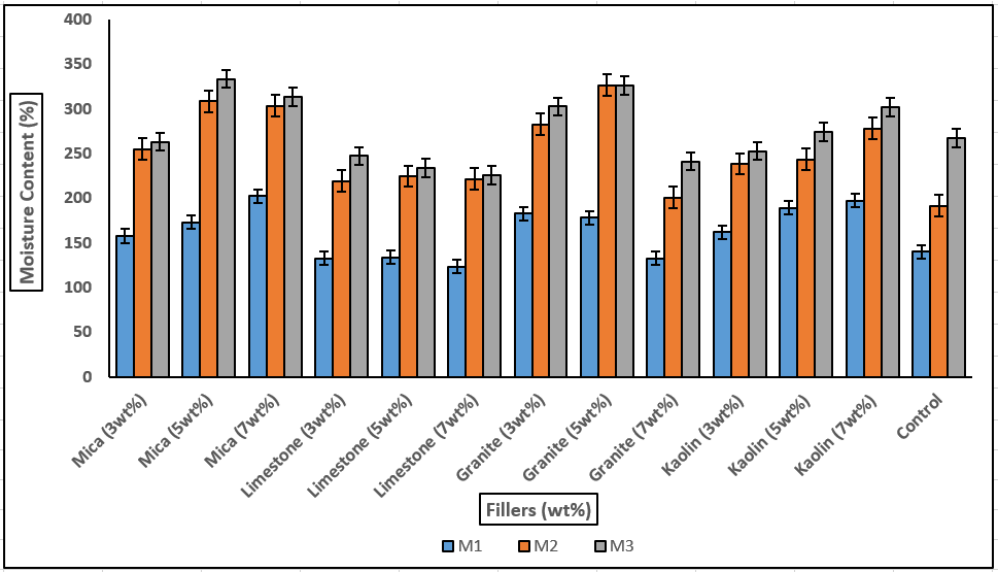


Fig. 10 *Water absorption of the filled & unfilled polyurethane foam.*

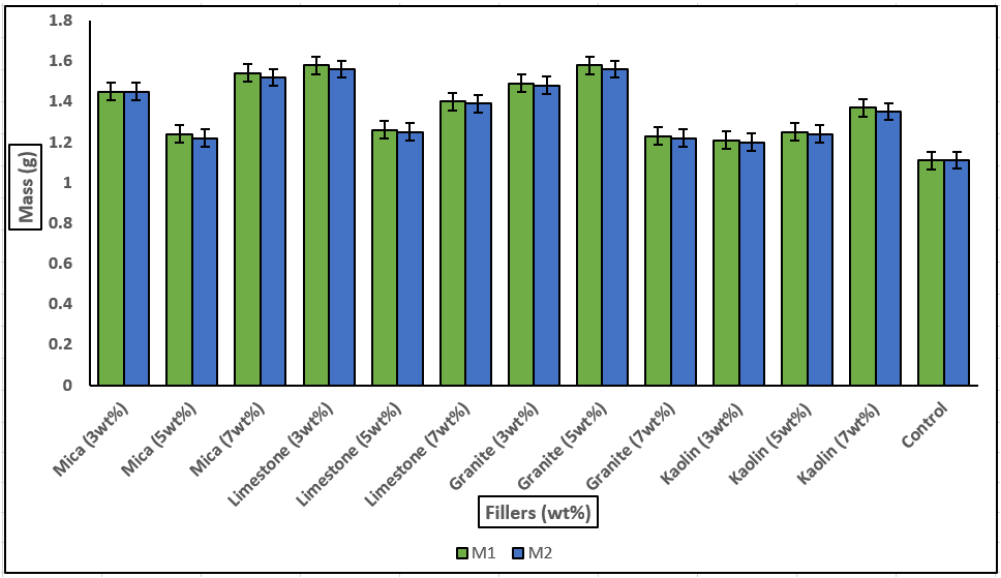


Fig. 11 *Thermal conductivity of the filled & unfilled polyurethane foam.*

4. Conclusion

Kaolin was discovered to be the most acceptable in improving the selected and evaluated mechanical properties of filled polyurethane foam. This ceramic filler gave the optimum enhancements in all the investigated mechanical properties and fell within acceptable limits in resistance to water absorption and thermal stability properties.

Kaolin-filled foam was the sample with close microstructural features to the unfilled polyurethane; hence, the one with the most improved properties. The enhancement was supported by its performance in the rheological properties as the optimum sample with desirable rheological properties.

The filler gave its optimum values at 5 wt.% for cream and gel times while it gave its minimum values for rise and the tack-free times at 3 wt.%. Aluminum and silicon were the elements that predominate the kaolin-filled polyurethane foam, which may be part of the reasons for the improved properties.

Conflict of Interest

There is no conflict of interest

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