



MICROSTRUCTURAL CHARACTERISATION, RHEOLOGICAL AND WATER ABSORPTION PROPERTIES OF FILLED POLYURETHANE FOAM

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Abstract

This work investigates the microstructural characterization, rheological and water absorption properties of granite and feldspar filled polyurethane foam. Foaming of polyurethane with the use of synthetic materials as suitable additives remain a major setback for polyurethane foams. Hence, granite and feldspar are selected as the fillers, pulverized and sieved to $<90\ \mu\text{m}$ and are randomly dispersed into the polyurethane matrix. 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 optimum fillers to be utilized in the production of rigid polyurethane foams given the effect of the fillers on the microstructural characterisation, rheological and water absorption properties of the foams. SEM, XRF and FTIR analysis were used to characterize the foams while rheological and water absorption tests are also carried out. The presence of the fillers in the foam showed a rupture in the structure of the foams with the cells having similar arrangements. The cream, gel, rise, and the tack-free time was delayed with the presence of the filler particles. It was discovered that 7 wt% feldspar addition enhances the cream and gel time while 3 wt% granite enhances rise and tack free time, respectively in the optimum conditions. It was discovered that the two fillers had inverse effects on the rheological properties. The addition of granite and feldspar degrades the sulfonic acid groups and promoted the appearance of Si-O stretching vibration band

Keywords: Ecofriendly, rheology, rigid foam, polymer composites, sustainable materials

Introduction

Polyurethane foam (PUF) is a widely used polymeric material in various applications and can be found virtually everywhere. Polyurethanes (PUs) happen to be one of the most important groups of polymeric materials due to their unique properties and vast areas of application (Gama *et al.*, 2018). Polyurethane foams are considered to have the largest consumption of PUs and amount to about 67% consumption of the total PU production (Czlonka *et al.*, 2020). While there are different types of polyurethane foams which are flexible PUF, rigid PUF, and semi-rigid PUF, rigid PUF is widely used in thermal and sound insulation applications as well as in structural and comfort applications. The different industries that require rigid polyurethane foams (RPUFs) for these applications include automotive, aerospace, packaging, construction, electronics, household appliances, marine, military, footwear, and furnishing industries (Gama *et al.*, 2018; Omotoyinbo *et al.*, 2021; Zhang *et al.*, 2014). RPUFs are known to possess a high

strength-to-weight ratio, high thermal stability, high toughness, good fire resistance, impact, and crack resistance (Gama *et al.*, 2018; Jabar *et al.*, 2018). They are considered the most efficient insulation materials because of their low thermal conductivity, hence, are majorly used for refrigerators, roof, and wall insulation (Kurańska *et al.*, 2019; Akkoyun and Akkoyun, 2019). Various research works have demonstrated the use of different organic and inorganic filler materials such as glass fiber, pulp fiber, talc powder, nanoclay, calcium carbonate, cellulose microfibrils, lignin, rapeseed, eggshell waste among others in time past to modify the functional properties of RPUF with emphasis on improving its mechanical, thermal and chemical properties (Omotoyinbo *et al.*, 2021; Agrawal *et al.*, 2019). This is crucial for RPUFs to have continued relevance for diverse and future applications. However, lesser research has been done in investigating the influence of these filler materials on the rheological properties of RPUF. This is likewise

important because the foaming process and the manufacturing procedure of RPUF largely affect the properties of the fabricated foam. A good understanding of the rheological properties of the foaming process of RPUF will enable the optimization of polymer chemical structure as well as the development of necessary technologies that help to produce desired cell morphologies (Jing, 2019; Kosmela *et al.*, 2019).

The science that deals with the deformation and flow of materials are known as rheology. In polymeric materials, the melt of a polymer is both elastic and viscous. This means that polymeric materials showcase both solid-like elastic behavior as well as fluid-like viscous behavior (Jing, 2019). The addition of solid filler materials to RPUF is expected to largely affect the viscosity hence the foaming process of the polyol blends (Strakowska *et al.* 2020). The formation and growth of cells in RPUF are dependent on the rheological properties of the polymer mixture which in turn determines to a large extent the mechanical and physical properties of the manufactured foam (Carrera *et al.*, 2010).

The use of mineral filler materials as reinforcements in polymers have been seen to provide low thermal expansion, good moldability and mechanical properties, high heat-deflection temperature, ecofriendly and low cost for the finished products foams (Oladele *et al.*, 2021a; Agrawal *et al.*, 2019; Oji and Oladele, 2019; Akkoyun and Akkoyun, 2019). Hence, this research investigates the influence of low-cost mineral fillers; feldspar and granite on the rheological and physical properties of RPUF. Feldspar is an alumina-silicate mineral that has a high resistance to abrasion, high chemical inertness, and low viscosity and is used as a filler material in the paint, adhesive, and rubber industries for improving the strength, durability, and toughness of the manufactured products (Agrawal *et al.*, 2019). Graphite occurs naturally and is a crystalline form of carbon. It is the most stable form of carbon under standard conditions. Expanded graphite which is made by immersing natural flake graphite in a bath of chromic acid and concentrated sulfuric acid can be used in the production of rigid polyurethane foams. Its thermal and electric conductivity helps to reduce heat dissipation and smoke production. It can be used as an effective flame retardant in RPUF and it enjoys large application in electronic products such as solar panels, electrodes and batteries (Strakowska *et al.*, 2020; Jabar *et al.*, 2020; Oladele *et al.*, 2019).

This research is therefore, carried out to investigate the influence of feldspar and granite fillers as mineral additives on the rheological and physical properties of rigid polyurethane foam. These fillers are selected as ecofriendly materials that can be used for foaming application instead of synthetic fillers that are commonly used presently but are not ecological and are of high cost.

Materials and Methods

Materials

Components A and B (pre-mixed with all the required additives) representing polyol and Isocyanate, respectively were procured from the Chemical and Allied Company in Lagos, Nigeria. Granite and feldspar filler materials were pulverized using the ball mill and then sieved to obtain a particle size of <90 µm. An unfilled polyurethane (RPUF) was first developed as a control sample followed by those with filler content. In each case, 60 g of components A and B were used while a varying proportion (3, 5, and 7 wt%) of each filler was used. The unfilled foam was developed by adding component B to A and mechanically stirring both components together for 10 seconds before being poured into an open mold. The filled foams were developed using a two-step process (Omotoyinbo *et al.*, 2021; Carrera *et al.*, 2010; Liguore *et al.*, 2005). In the first step, each of the filler in varying proportions was added to component A and dispersed evenly by a mechanical stirrer. In the second step, component B was added to the existing mixture and thoroughly stirred together for 10 seconds before being poured into a mold to allow for free foaming in the vertical direction (Kim *et al.*, 2008). During the stirring and foaming process of each sample, rheological properties; such as; cream time, gel time, rise time, and tack-free time of the samples were recorded using a digital stopwatch. The dimension of the molds used was 8×10×13 cm. Each of the foam was de-molded after 30 minutes and was allowed to cure at room temperature for 24 hours. Finally, a dimension of 3 cm³ was cut out from each sample and the required property tests were carried out.

Microstructural Analysis

Microstructural analysis of the unfilled and granite/feldsparfilled polyurethane foams was done using field emission scanning electronic microscope (JEOL JSM-760 0F). The accelerating voltage of the microscope is 15 kV. Prior to the analysis, the samples were gold sputtered to improve electrical conductivity.

Water Absorption

Water absorption test was carried out on all sectioned samples of the filled foam and the controlled samples for 96 hrs. After immersion in distilled water for 24 hours at an ambient temperature of 24 ± 2 °C, the test samples were removed from the water and surface liquid wiped off using a dry cloth before immediately weighing. A digital scale was used to take the initial (w_1) and final (w_2) weights of the test samples to the nearest 0.0001 g, afterwards, the test samples were returned to the water for continuing exposure. The moisture content of the foams was determined using Equation 1.

$$\text{Moisture content} = \frac{w_2 - w_1}{w_1} \times 100 \quad (1)$$

XRF Analysis

X-Ray Fluorescence (XRF) analysis was carried out on the granite and feldspar based polyurethane foams to determine their elemental compositions. The chemical analysis was performed with Energy Dispersive Spectrometer (EDX3600B). About 0.5 g of the sample material was pressed using lithium tetraborate (flux) which allow evenly dispersed solid solution. The x-rays beamed on the sample and cause an excitement on some of the atomic nuclei to jump to the higher energy orbitals to facilitate the identification of the elements in the sample.

Fourier Transform Infrared Spectrometer (FTIR) Analysis

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 resulting spectra from them 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 4 cm^{-1} . The infrared spectrum was Fourier transformed and recorded in the absorption mode.

Results and Discussion

Microstructural analysis of the Polyurethane Foam

Figure 1 presents the structure and cell sizes of the unfilled polyurethane, feldspar and granite filled foams is shown in Figure 1(a-c) respectively. The foams are closed cells with their average cell sizes ranging between 100 - 200 μm . The presence of the fillers in the foam showed a rupture in the structure of the foams with the cells not being uniform. The granite and feldspar spheres are distributed uniformly in the polyurethane matrix. The interface of the foam and particles is continuous with the particle surface having a high degree of roughness and limited porosity. The degree of roughness and porosity are helpful features that promotes bonding of the particles with the matrix.

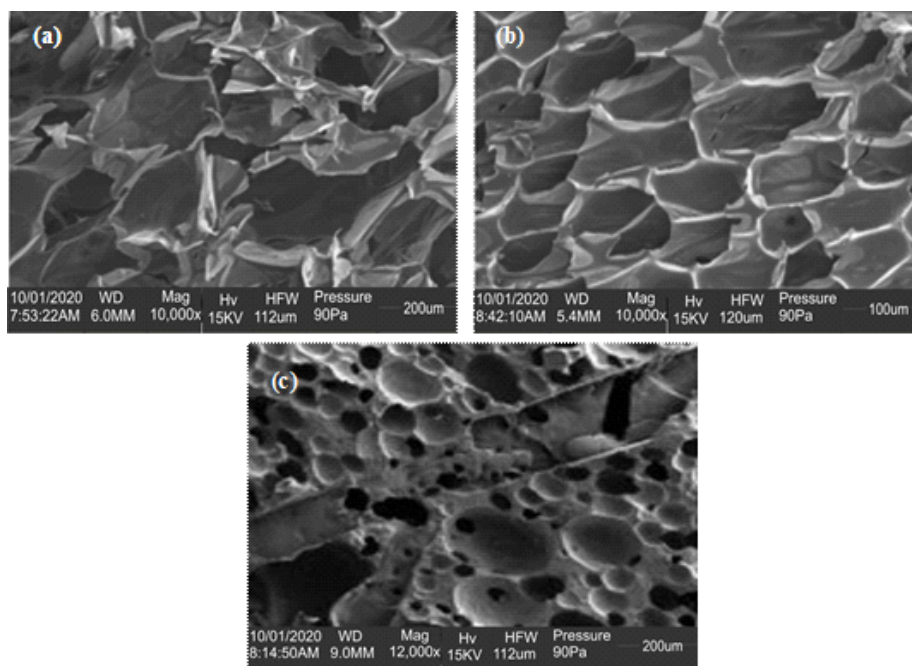


Figure 1: SEM micrographs of samples (a) Unfilled polyurethane foam (b) feldspar filled polyurethane foam (c) granite filled polyurethane foam

3.2 XRF Analysis of the Filled Polyurethane Foam

XRF analysis was carried out for the fillers in order to determine the elemental composition present. Figure 2 and Table 1 show the peaks and the corresponding values, respectively for granite filled foam. From the results, it was observed that silicon and iron were the

predominant elements with values of about 20% and 9%, respectively. Other elements that are predominant are: potassium (7%), aluminium (4%) and calcium (3%). This findings was in agreement with the work by Omotoyinbo *et al.* (2021). These elements may likely enhanced the properties of the developed foam.

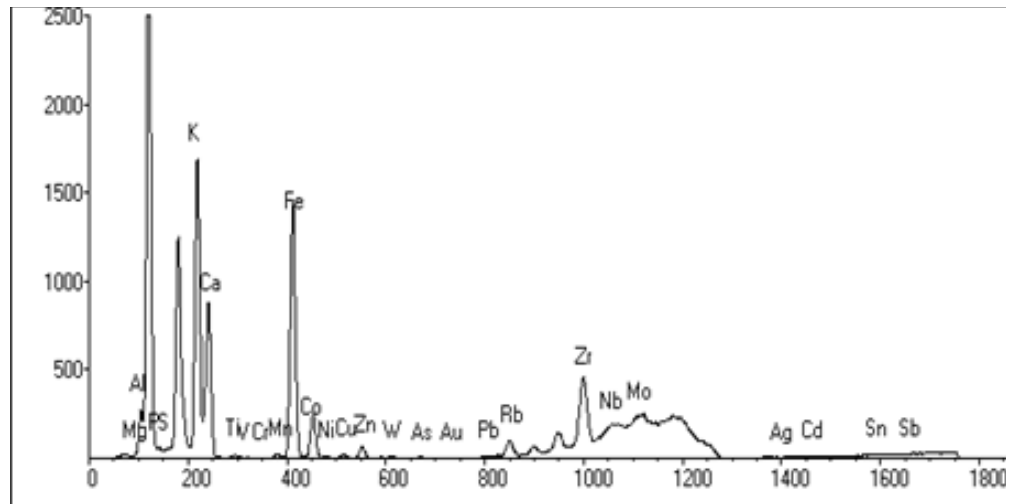


Figure 2: XRF peaks for granite granite filled polyurethane foam

Table 1: XRF Result for Granite Filled Polyurethane Foam

Element	Intensity	Content
Al	0.0151	3.8156
Si	0.1821	20.2382
P	0.0049	0.2282
S	0.0080	0.6026
K	0.0812	6.6192
Ca	0.0454	3.4429
V	0.0002	0.0098
Cr	0.0002	0.0085
Mn	0.0017	0.1153
Co	0.0030	0.1683
Fe	0.0915	8.7753
Ni	0.0008	0.0435
Cu	0.0015	0.0330
Zn	0.0045	0.1531
Rb	0.0070	0.0677
Nb	0.0046	0.0524
Mo	0.0046	0.1552
Sn	0.0061	1.1422

Also, Figure 3 and Table 2 show the results of the XRF for feldspar filled foam where it was observed that silicon and aluminium were the predominant elements.

While silicon was about 42%, aluminium was about 18%. It was discovered that for both granite and feldspar, silicon was the element with highest content.

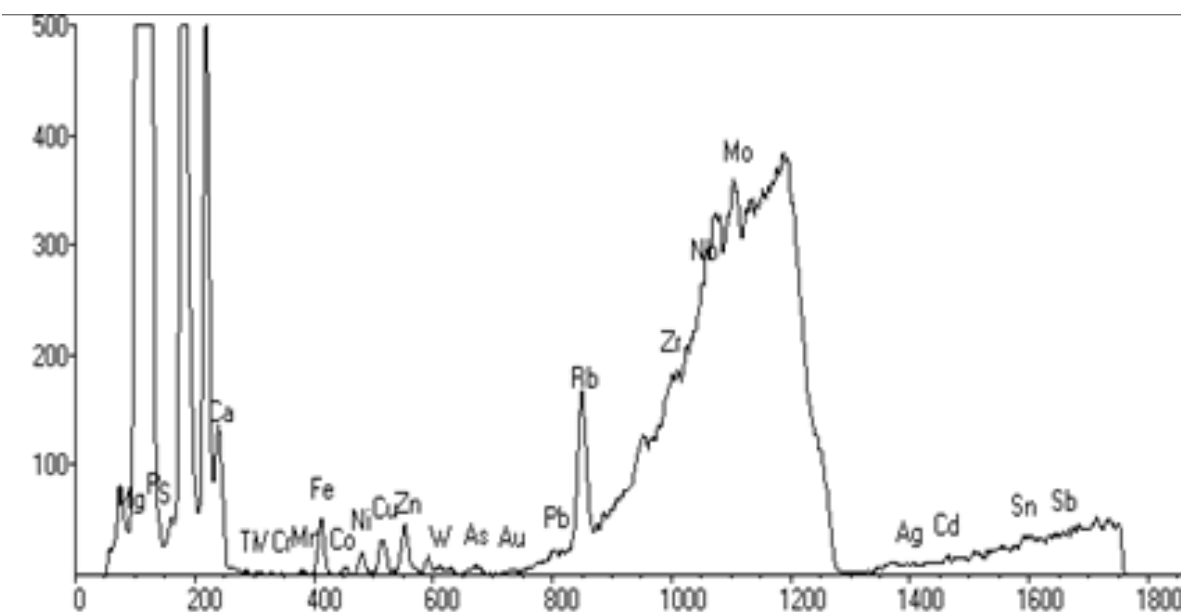


Figure 3: XRF peaks for feldspar filled polyurethane foam

Table 2: XrfResult for Feldspar Filled Polyurethane Foam

Element	Intensity	Content
Al	0.0879	17.8466
Si	0.3364	41.7202
P	0.0042	0.1974
S	0.0045	0.3006
K	0.0185	1.5011
Ca	0.0048	0.0687
V	0.0001	0.0027
Fe	0.0024	0.3109
Ni	0.0010	0.0599
Cu	0.0019	0.0468
Zn	0.0026	0.0861
Au	0.0002	0.1218
Mo	0.0024	0.2215
Sn	0.0071	1.3216
Sb	0.0097	1.2093

Rheological Properties

Rheological properties of a material are the properties that have to do with the flow of the material when exist in liquid state which have linear influence on the material when turn to solid (Oladele *et al.*, 2011). The foaming process of the RPUF composites was monitored by measuring the different processing times, which are cream time, gel time, rise time, and tack-free time using a digital stopwatch (Suleman *et al.*, 2014). The time taken for each foaming process is presented in Figure 4.

Cream Time

In the production of the rigid foam, the cream stage is the first reaction stage which occurs from mixing the reactants (polyol, isocyanate and filler). The cream time is the time taken in seconds for the reactants to attain a homogenous mix and produce a milky or cream-like color. The cream time of the samples varied and depend majorly on the quantity of the constituents used. From the results, it was discovered that the cream time increases as the filler content increases for granite addition while it decreases as the filler content increases for feldspar addition. Sample with 7 wt.% feldspar addition had a cream time of 9 s next to the control sample that has the least cream time of 3 s. This implies that there is a delay in the chemistry of the polyurethane with the addition of the fillers.

Gel Time

This is characterized as the period when bubbles are being formed or when the reaction starts foaming or rising. Similar trend with cream time was observed for the gel time. From the results, it was noticed that sample with 7 wt.% feldspar addition had a gel time of 28 s close to the control sample that has the least gel time of 14 s. The addition of fillers to the polyurethane (PU) increases the viscosity and affects the foaming process (Muhazeli *et al.*, 2019). The gel time of the filler induced PU is increased when compared to that of the unfilled foam. The samples achieve a gel state after the homogenous mix begins to form into a pasty substance. The time taken for the various samples to gel directly

affects their rise time. The evenly dispersed filler particles tend to act as extra nucleation centers leading to the formation of more bubble cells with reduced cell size during the nucleation process thereby increasing the gelation period of the foam (Widya and Macosko, 2005).

Rise Time

After the gelling of the PU foams, it then begins to expand under free rise as a result of continuous stirring which leads to the production of the rigid polyurethane foams. On average, the rising of the foam is almost instantaneous but the influence of some of the fillers brings about variations in the rise time. Similar trends to the other variables were observed but the least rise time was obtained from 3 wt% granite addition with a value of 56 s close to the control sample of 52 s. The faster the foam can reach full rise, the faster it is able to cure which happens when all the physical properties of the foam are completely established within the foam.

Tack Free Time

After the foam attains full rise, the surface of the foam remains sticky and goes off as the foam gradually cures. This sticky nature can be observed by touching the surface of the foam. The rise time of the various samples directly affects the time taken for the sample to become completely tack-free as the faster the rise time, the faster the sample becomes tack-free which in turn results in a faster curing time of the foam. The time taken for the PU foams to become tack-free varied with the filler content addition in a similar mode to that of rise time. It was noticed that the least tack free time was obtained from 3 wt% granite addition with a value of 63 seconds close to the control sample of 54 seconds. Hence, these two fillers influence the rheological properties in different ways. While feldspar was highly effective in cream and gel times at higher filler content (7 wt%), granite was effective for rise and tack free times, respectively at low filler content (3 wt%). Since, these parameters are inseparable, there may be need to consider hybrid filler or the addition of higher/lesser proportions of the individual fillers for more improved rheological properties.

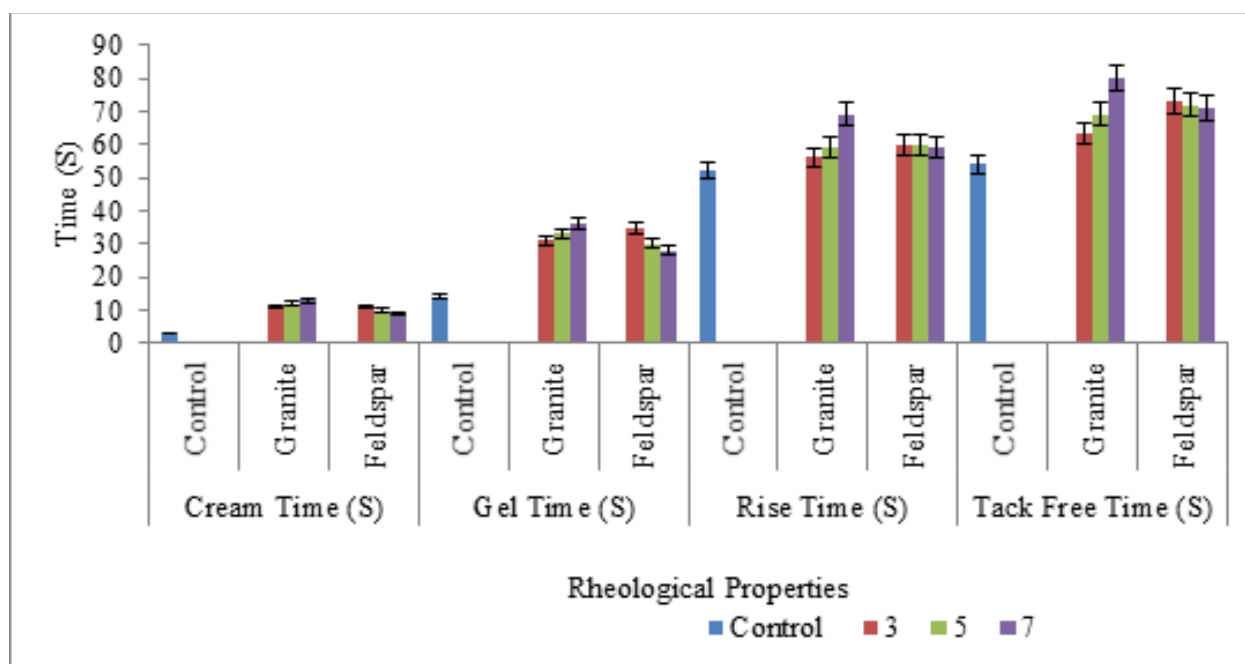


Figure 4: Rheological properties of the developed feldspar and granite reinforced polyurethane foam

3.4 Water Absorptivity

The effect of water absorption rate on the developed foams were presented in Figure 5. From the results, it was showed that water absorption rate increases as the filler content increases. The results showed that granite based foam absorbed more water than feldspar based foam and the control in all the filler content considered. On the other hand, feldspar-filled foam absorbed less water within 3-5 wt% but absorbed more than the control sample at 7 wt%. The high absorption obtained at high filler content may be due to less interfacial

adhesion due to higher filler content (Oladele *et al.*, 2021b; Alaneme *et al.*, 2013). Hence, the addition of feldspar within 3-5 wt% aid hydrophobicity of the foam and this can be applicable in an area where moisture absorption is the major environmental challenge to be faced by the foam. The maximum amount of water absorbed after 72 hrs was 148 and 213 g, respectively for 3 and 5 wt% feldspar addition compared to 268 g for control sample. Thus, this filler can be used for the development of foam for use in boats and other marine applications.

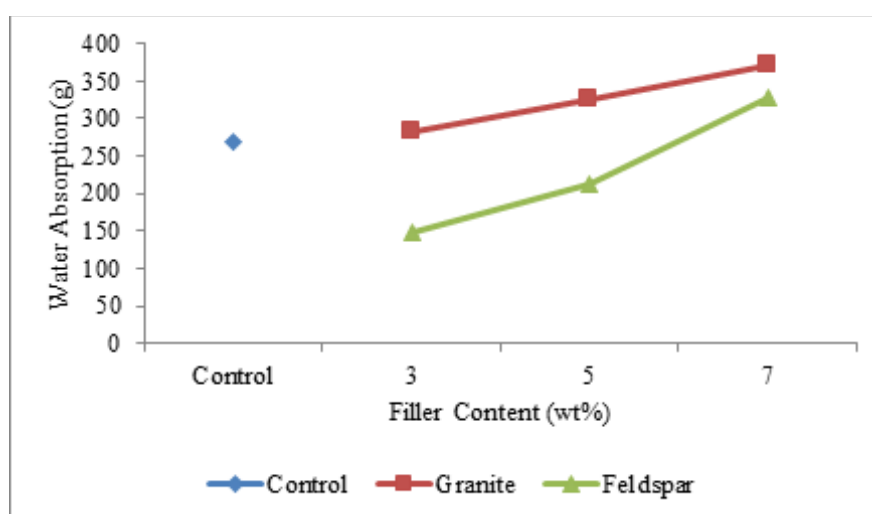


Figure 5: Water absorption properties of the developed feldspar and granite reinforced polyurethane foam and the control after 72 hrs

Fourier Transform Infrared Spectrophotometer Analysis

Fourier Transform Infrared Spectrophotometer (FTIR) is a powerful tool for identifying the types of chemical bonds (functional groups) present in compounds. The FTIR measurement was used in this study to examine the changes in composition and chemical functional groups in the granite and feldspar filled polyurethane foam. Figures 7-9 and Tables 3-5 depict the FT-IR spectra and data of the unfilled polyurethane foam (Fig. 7; Table 3), polyurethane foam filled with granite particles (Fig. 8; Table 4) and polyurethane foam filled with Feldspar particles (Fig. 9; Table 5) with significant functional groups (wavelength between 4000 cm^{-1} – 1500 cm^{-1}) labeled for clearness. The polyurethane foam without fillers (Fig. 7; Table 3) revealed two O–H groups responsible for the stretching vibration and stretching at 3098 and 2910 cm^{-1} , respectively. The foam also shows a clear C–O–C peak at 1099 cm^{-1} with chain extenders consisting of the sulfonic groups (Jabar et al. 2018). The sulfonic groups S–H, S=O and O–S–O peaks appear at ~1100, 1398 and 1050 cm^{-1} , respectively with the O–S–O peak partially

overlapping the C–O–C band. The urethane carbonyl peaks and H–NH band also appeared with different intensities as reported in Table 3. The –CH bending vibrations due to substitution groups in alkenes and aromatics also appeared at 926 cm^{-1} . The different peaks that appeared in the unfilled foam is an indication that both functional and sulfonic acid groups were successfully integrated into the polyurethane backbone. It is clearly observed that the addition of granite and feldspar as presented in Figures 8 and 9 causes a change in modification of the absorption peaks. The sulfonic acid groups present in the unfilled polyurethane foam were observed to be absent in the granite and feldspar filled polyurethane foams. The degradation of the sulfonic acid groups could be related to the high content of silica in the granite and feldspar which promoted the appearance of Si–OH and Fe–O–Si stretching vibration band, respectively. Amorphous Na and K linked to Oxides were also noted to be present in the granite filled polyurethane foam.

Table 3: FTIR Spectrum Values for Unfilled Foam

Run #	Peak (cm^{-1})	Transmittance (%)	Absorbance	Assignment
1	3098.22	61.35	0.2122	O-H Stretching vibration
2	3095.11	48.20	0.3170	-C=C- stretching
3	3043.40	28.94	0.5385	H-NH stretching
4	2910.25	16.47	0.7833	O-H stretching
5	2834.02	48.11	0.3178	C-H stretching vibration of polyol
6	1942.61	85.73	0.0669	C=N stretching
7	1710.26	81.35	0.0896	S-H stretching
8	1608.71	52.41	0.2800	CH ₂ asymmetric variation
9	1498.37	20.03	0.6983	deformation CH ₂ + C=C of the aromatic ring
10	1450.11	20.71	0.6838	C-H bending of unsaturated methylene groups.
11	1398.47	73.95	0.1311	S=O asymmetric Stretching
12	1099.20	73.04	0.1364	C-O-C asymmetric stretching
13	1050.22	64.39	0.1912	O-S-O stretching
14	926.34	72.16	0.1417	-CH bending vibration to substitute in alkenes and aromatics
15	771.42	28.74	0.5415	-CH ₂ bending
16	708.15	4.10	0.3872	C=C aromatic stretching
17	528.73	60.41	0.2188	C-Cl stretching vibration

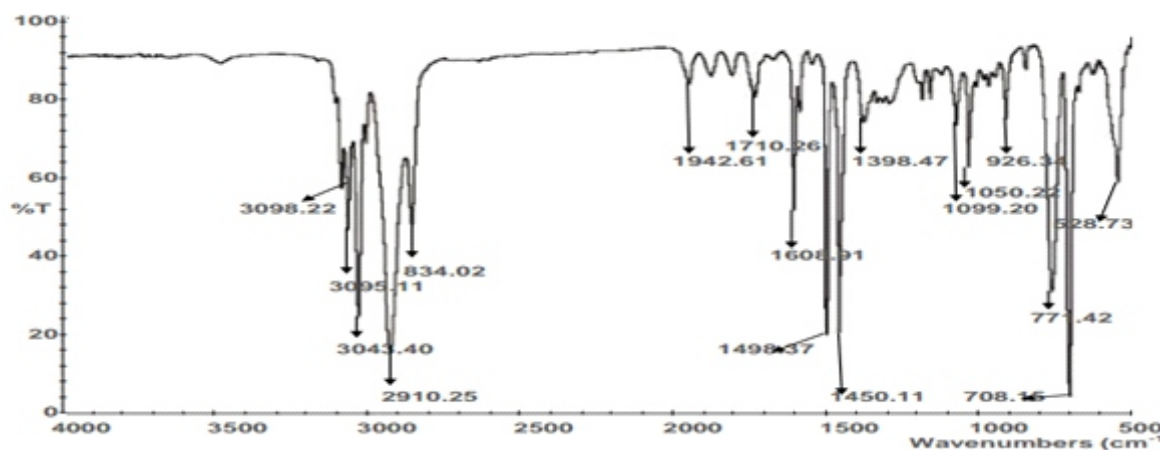


Figure 7: FTIR spectrum for unfilled polyurethane foam

Table 4: FTIR Spectrum Values for Granite Filled Foam

Run #	Peak (cm ⁻¹)	Transmittance (%)	Absorbance	Assignment
1	2480.15	84.21	0.0746	O-H Stretching
2	2381.07	22.61	0.6457	N-H Stretching
3	2312.63	40.03	0.3976	C-H stretching
4	1708.36	96.22	0.0167	Si-OH stretching mode in quartz
5	1214.39	16.53	0.7817	Amorphous Na and K linked to Oxides
6	963.21	65.02	0.1670	C-O-C asymmetric stretching
7	920.83	75.82	0.1202	Organic-inorganic hybrid bond
8	861.03	61.26	0.2128	-C=C-stretching
9	847.25	35.18	0.4537	N-H bending
10	800.41	56.14	0.2507	Si-OH stretching
11	736.11	84.97	0.0709	Al-O stretching
12	682.18	52.61	0.2789	N-O symmetric stretching
13	610.31	83.50	0.0783	C-Cl stretching vibration
14	574.24	83.66	0.0774	-OH deformation

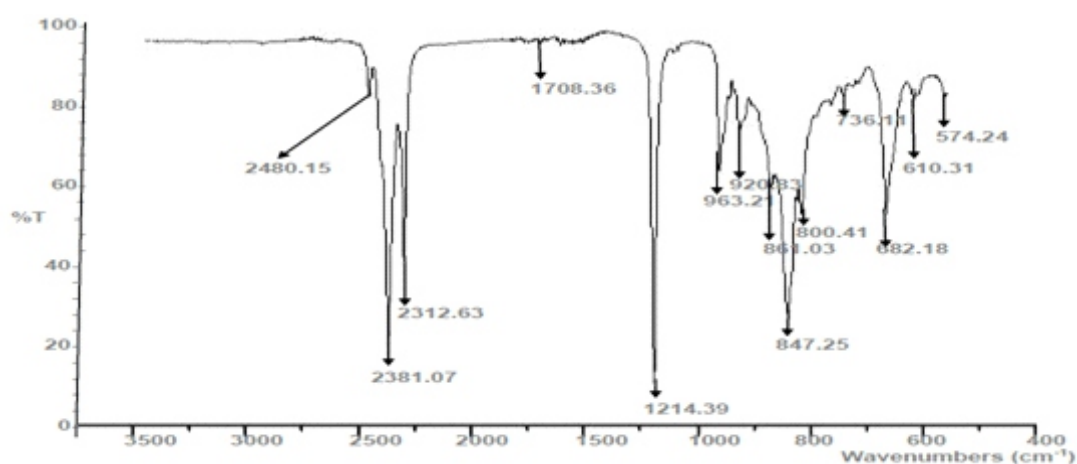


Figure 8: FTIR spectrum for granite filled polyurethane foam

Table 5: FTIR Spectrum Values for Feldspar Filled Polyurethane Foam

Run #	Peak (cm ⁻¹)	Transmittance (%)	Absorbance	Assignment
1	2420.41	8.20	1.0862	-NH ₂ Stretching vibration
2	2307.36	24.10	0.6180	C-H Stretching
3	1724.14	84.31	0.0741	-NH ₂ Stretching vibration
4	1212.66	28.10	0.5513	C-N-C stretching vibration
5	991.65	48.69	0.3126	C-O-C asymmetric stretching
6	920.68	60.10	0.2211	-NH deformation
7	889.34	44.18	0.3548	Hydrogen bonding of N-H stretching
8	856.21	25.03	0.6015	Asymmetric vibration of CH ₂
9	815.33	44.82	0.3485	Symmetric vibration of CH ₂
10	761.20	64.57	0.1900	C- N stretching
11	732.21	64.76	0.1887	C=C aromatic stretching
12	681.30	54.15	0.2664	Fe-O-Si stretching vibration

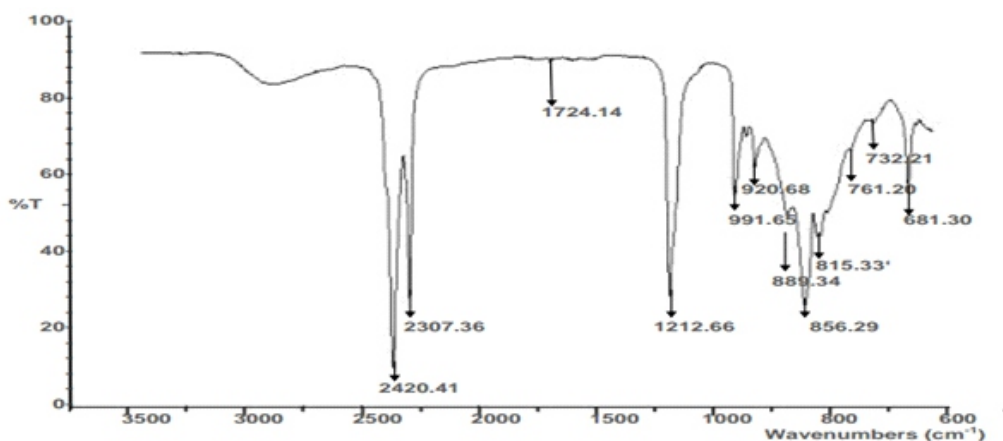


Figure 9: FTIR spectrum for feldspar filled polyurethane foam

Conclusion

This study has shown that the addition of fillers in the production of polyurethane foam enhances the microstructure, rheological and water absorption properties of the foams. The cream, gel, rise, and the tack-free time was delayed with the presence of the filler materials. It was discovered that 7 wt% feldspar addition enhances the cream and gel time while 3 wt% granite enhance rise and tack free time, respectively in the optima conditions. It was discovered that the two fillers had inverse effects on the rheological properties. Feldspar was able to aid hydrophobicity in feldspar-filled polyurethane foam. The addition of feldspar and granite to polyurethane foam causes a change in modification of the absorption peaks with the degradation of the sulfonic acid groups.

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