DOI: 10.36108/ujees/3202.50.0130
UNIOSUN Journal of Engineering and Environmental Sciences, Vol. 5 No. 1. March 2023

# Microstructural Properties of Gypsum Composites containing Pineapple Leaf Fibre

**Esan, M. T., Khairulzan, Y., Zaiton, H., Gambo, M. D. and Hassan, H.**

**Abstract:** The interest in prospective uses of gypsum as a finishing component has diminished in recent years as a result of its weak mechanical strength, which is essential in interior construction. Therefore, to overcome this challenge, researchers have come up with various ways of improving the gypsum composite materials. The microstructure of the composites and the contact surface with pineapple leaf fibre were investigated by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). The microstructural properties of the gypsum paste changed when pineapple leaf fibre was added. These studies showed that at 2% fibre volume, a jellylike surface coating was formed around the gypsum crystals, giving the pineapple leaf fibre (PALF)-gypsum composite its high interlocking and compactness, thereby impacting high strength than the pure gypsum and this suggests its broader application in building industry for production of partition boards.

**Keywords:** Gypsum; Microstructure; X-Ray Diffraction (XRD); Scanning Electron Microscopy (SEM); Pineapple leaf fibre.

## I. Introduction

Gypsum is a binding material created by heating and partially or completely eliminating the crystallization water that is naturally present in gypsum rock. It is one among the first materials that people used for building. Depending on the temperature, heating calcium sulphate dehydrate ($CaSO_4 \cdot 2H_2O$) from gypsum rock causes partial or total dehydration. It can be transformed into anhydrite or hemihydrate ($CaSO_4 \cdot 0.5H_2O$) [1]. Gypsum rock entirely dehydrates when heated to temperatures over 190–200 °C. The water that causes the gypsum rock to crystallize is lost in this circumstance. This results in $CaSO_4$, commonly known as gypsum anhydrite [2], [3].

**Esan, M.T., Khairulzan, Y., Zaiton, H.**
(Department of Structure and Materials, School of Civil Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia)
**Esan, M.T., Gambo, M.D. and Hassan, H.**
(Federal Polytechnic, Ede, Nigeria.)
**Corresponding Author:**
**Tel.: +234-8038194026; Email: esanmartins@yahoo.com**

Depending on the method of calcining used during production, the hemihydrate can have one of two distinct crystal structures: alpha, which is a saturated atmospheric water vapour, when made from gypsum using a wet method, and beta, which is a saturated atmospheric water vapour created using a dry method. Unprocessed gypsum, which is composed of calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$), is chemically converted into calcium sulfate hemihydrate ($CaSO_4 \cdot 0.5H_2O$) by calcining by eliminating 1.5 water molecules [4]. The alpha-hemihydrate is a building material that is referred to as "Plaster of paris" [5]–[7]. The hydration of hemihydrate has been the subject of numerous investigations, including those on the kinetics and setting of hydration [8]–[13]. Equations 1 and 2 combined to represent the integral reaction to hydration. Equations (2) and (3) show how hemihydrates quickly hydrate after combining with water [14], first becoming

water-soluble (Equation (2)), then becoming anhydrous gypsum (Equation (3)).

$CaSO_4.0.5H_2O \rightarrow Ca2^{+} + SO_4^{2-} + 0.5H_2O$ (1)

$Ca^{2+} + SO_4^{2-} + H2O \rightarrow CaSO_4.2H_2O$ (2)

$CaSO_4.0.5H_2O + 1.5H2O \rightarrow CaSO_4.2H_2O$ (3)

$Ca^{2+}$ and $SO_4^{2-}$ are produced when the hemihydrate dissolves in water; when they combine, a dihydrate is produced, which precipitates out of the solution. As a result, crystallization nuclei appear, and crystals develop around them to form a network of long, interconnecting crystals that gives the substance its strength [10], [14], [15]. This hydration mechanism is explained by Le Chatellier. The resulting compound, $CaSO_2.2H_2O$, shares the same chemical composition as gypsum rock [16], [17]. During the hydration phase, the hardened paste's microstructure is established. It is determined by the water to gypsum ratio, which is responsible for initiating hydration processes and ensuring the workability of the gypsum slurry. Gypsum crystals become looser and create vacancy fractions as the amount of water in the substance rises, which lowers its strength [6], [10]. In comparison to other kinds of inorganic cement, the gypsum binder is thought to be more ecologically friendly because of its low thermal treatment temperatures and capacity for closed-loop recycling [1]. At the moment, wall coverings, partition walls, and ceiling designs are the main uses for gypsum plasters. Gypsum-based products, including as mortar, composites, and boards, are being utilized in construction more and more frequently because of their superior fire resistance, thermal properties, and sound insulation advantages [18]–[27].

Gypsum board is growing in popularity because of its excellent potential for sound insulation, speed of construction, and ease of installation. Contrarily, plain gypsum board is fragile, has a low flexural strength, and has poor sound absorption [28]–[30]. The physical and mechanical properties of gypsum products have been improved by scientists using a variety of additive-based approaches. In addition to expanded polystyrene (EPS), silicate fillers (clay minerals, perlite, vermiculite, silica fume, fly ash), and foaming agent additions, other appropriate choices include glass fibres, mineral wool, cork, organic fibre and rice husk [31]. In the literature, fillers such as rice husk [32], blast furnace slag (BFS) [33], nanotubes [33], iron oxide [34], calcium carbonate [32], silica gel [35], and silica and fume [35] have been employed in gypsum-based composites. Fibre, which normally has a higher tensile strength than the gypsum matrix, appears to help increase the mechanical effectiveness of the composite by spreading the applied stress load [36], [37]. Fibreglass and natural fibres were employed in earlier experiments to solve this issue [38]–[40]. Fibres were added to the gypsum board to increase its mechanical properties. The fibres act as reinforcements, reduce the matrix's brittleness, and improve how evenly the matrix is stressed [41]. The quantity of trash produced as a result of the quick setting time of the gypsum presents another issue with the usage of gypsum products [15], [42]. However, despite recent efforts, the development of composite materials still confronts a number of challenges, many of which need solutions. As a result, it is necessary to develop gypsum composites with natural fibres,wich reduce potential for global warming caused by carbon dioxide emissions from the production of various synthetic fibres that

has been used before in making composite materials.

Pineapple leaves can also serve to reduce environmental consequences, because they are frequently discarded and constitutes wastes. As a result, the focus of this study is on the microstructure properties of the PALF-gypsum composite. The aim of the study has lately been impacted by the quest for ecologically friendly building materials. The study's conclusions will be beneficial to the building sector, which calls for the adoption of more lightweight materials.

## II. Materials and Methods

### A. Materials

Gypsum plaster, which is commonly accessible at the market, was the type of natural gypsum employed in this experiment. Table 1 provides the gypsum's chemical composition. Ground PALF as shown in Figures 1 was purchased from Chemical Engineering department, Universiti Teknologi Malaysia. Table 1 and 2 provides a list of the chemical characteristics of the gypsum and PALF used in this experiment. In Figures 2 and 3, the particle size distributions of the PALF and gypsum powder employed in this investigation are displayed.

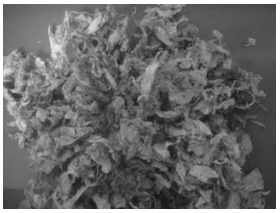


**Figure 1: Pineapple fibre with 15mm size**

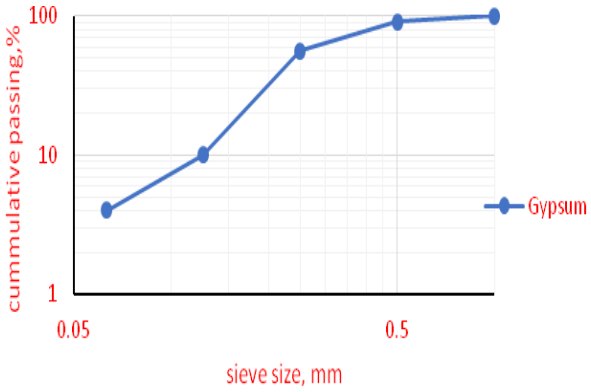



**Figure 2: Gypsum Particle Size Distribution**

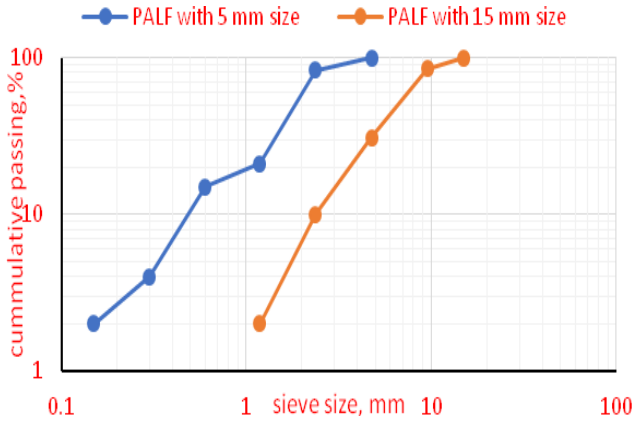



**Figure 3: PALF Particle Size Distribution**

The chemical composition of gypsum is shown in Table 1. It contains the oxides of several metallic elements. The major component of gypsum is Sulphur trioxide ($SO_3$). The mass content of Sulphur trioxide was 52.1%. This is the main oxide components that contributes to secondary hydration in the gypsum matrix as in Equation 2. In addition to the main components, a substantial amount of lime (CaO) is also present, about 46.8%. There were other oxides such as magnesia (MgO) with 0.222%, silica ($SiO_2$), Iron oxide ($Fe_2O_3$) and Alumina ($Al_2O_3$) in reasonable

quantities. The oxides, such as MgO, $Al_2O_3$, $Fe_2O_3$ and $SiO_2$, shows no significant content. It shows there are very small impurities in the sample. According to [43] the minimum content of $CaSO_4.2H_2O$ in gypsum for building use should have at least 70% by weight, and this can be obtained by summing up the value of $SO_3$ and CaO. As can be seen from Table 1, the gypsum rock content of $S0_3$ and CaO planned for use in the investigation was 52.1% and 46.8%. Which implies the value of $CaSO_4.2H_2O$ was 98.25%. The composition and percentage of chemical composition of gypsum may vary depending on their origin. According to the XRF results, the gypsum had similar chemical compositions compared with other gypsum reported in the literature [44]; [45]. The gypsum consists essentially of sulphur trioxide and lime with other components in smaller amounts.

**Table 1: Gypsum chemical composition analyzed by XRF**

| **Chemical composition (Oxide)** | **Lime (CaO)** | **Sulphur trioxide ($SO_3$)** | **Silica ($SiO_2$)** | **Alumina ($Al_2O_3$)** | **magnesia (MgO)** | **Iron oxide ($Fe_2O_3$)** |
|---|---|---|---|---|---|---|
| Mass content (%) | 46.8 | 52.1 | 0.517 | 0.354 | 0.222 | 0.0433 |

The chemical composition of PALF is detected using XRF analysis and presented in **Error! Reference source not found.**2. From the table, it is noticed that Lime (CaO) and Sulphur trioxide ($SO_3$) are the most dominant oxides in the sample with mass content of 55.9% and 35.4% respectively. There were other key oxides such as magnesia (MgO), Alumina ($Al_2O_3$), Silica ($SiO_2$) and Iron oxide ($Fe_2O_3$) in reasonable quantities. The chemical attributes under investigation by other researchers were on cellulose, hemicellulose, holocellulose and lignin, apart from the study conducted by [46] which also studied the chemical composition (oxide) of PALF. Although the results obtained from this study was not similar with the results in this present study, the result revealed the presence of silica, carbon, oxygen and nitrogen on the treated and untreated PALF. This is due to different treatments applied on PALF, sources of fiber and process of obtaining the fiber. For example, in this study 6% NaOH solution was used in the treatment of PALF while the combination of 5% silane and NaOH concentration were applied for the PALF treatment according to the study conducted by [46].

**Table 2: PALF chemical composition analysed by XRF-**

| **Chemical composition (Oxide)** | **lime (CaO)** | **Sulphur trioxide ($SO_3$)** | **Silica ($SiO_2$)** | **magnesia (MgO)** | **Iron oxide ($Fe_2O_3$)** | **Alumina ($Al_2O_3$)** |
|---|---|---|---|---|---|---|
| Mass content (%) | 55.9 | 35.4 | 3.01 | 2.9 | 1.74 | 0.982 |

### B. Preparation of the PALF-Gypsum Composite Material and instrumentation

In this study, the gypsum composite was created using gypsum, PALF, and tap water. A 0.6 water-to-gypsum ratio was used to create 15 mm fibre diameters for various PALF percentages. The composite PALF-gypsum mixes were made using a constant water-gypsum ratio of 0.6. The ratio was determined based on trial mix results from earlier tests, which showed that PALF-Gypsum with a 0.6 water-gypsum ratio had exceptional workability. To compare the samples, a set of controls that lacked PALF was also created. Then, C2, C3, C5, C10, and C20 were substituted for the PALF increases of 2%(C2), 3%(C3), 5%(C5), 10%(C10), and 20%(C20), respectively.

In order to ascertain the mechanism of strength increase with and without PALF, the morphology of the gypsum composites was examined under a scanning electron microscope. The chemical composition of the PALF and gypsum were identified using X-ray fluorescence. The diffraction pattern of gypsum with and without PALF was also measured using an X-ray diffractometer with the model number Rigaku D-max 2200.

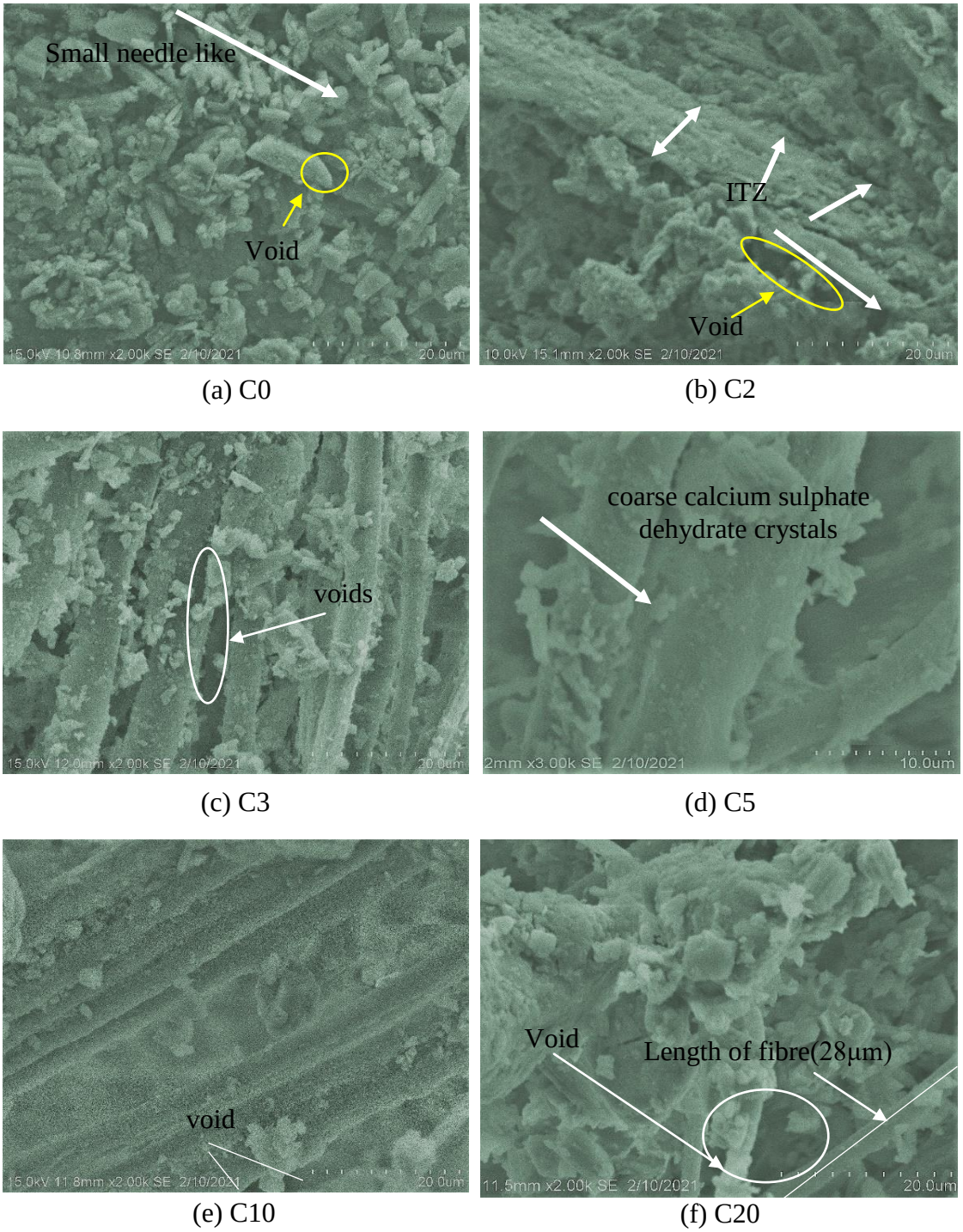
## III. Results and Discussion

### A. Scanning electronic microscopy

Figure 4 displays the 15 mm PALF-gypsum composites' micrographs. It was shown that PALF altered the microstructure of the gypsum matrix. The micrograph of the control specimen shows a small, needle-shaped crystal with a length of 11.31 μm. Similar observations were made on the SEM micrograph of fibre according to study conducted by [47]. Additionally, it might be challenging to locate the pores (as shown in Figure 4a control). The crystal's diameter is 3.81 to 3.9μm, while the pore size is around 27μm. However, as seen in Figure 4b, the needle-like crystal lengthens when PALF is administered. According to [48] the control hydrates significantly and the induction and acceleration durations are short. The crystal then struggled to develop into a long, thick crystal. However, in the case of PALF-gypsum composites, these specimens experience lengthy induction and acceleration times, which are followed by lengthy crystal development processes that cause the crystal to become long and thick. The gypsum particles are tightly bound to the PALF, as seen by micrographs of the composite PALF-gypsum material. This may be demonstrated with PALF volume fractions of 2%, 3%, and 5%. The interfacial transitional zone (ITZ) was compact with a small gap after adding 2% PALF to the gypsum matrix. The diameter thickened to 4.1μm, the crystal length increased to 40.42m, the pore size decreased to 60m, and the crystal with 3 percent PALF had a longer length (32.27m) and a wider diameter (5.93μm) as shown in Figure 4c. [42] also observed a similar trend in their work when looked into the mechanical properties of cellulose fiber, the surface adhesion between the fibres and the gypsum matrix was improved when recycled cellulose pulp was employed. The composites' mechanical qualities improved as a result.

The existence of voids in the composites is shown by micrographs taken at higher fibre volume percentages. The occurrence of

voids between fibres for the 5% fibre (14m) suggests that composite delamination, with the separation contact along the PALF, was the primary cause of failure. A 10% increase in PALF concentration (Figure 4e) resulted in a 47m increase in crystal length and a 99m pore size on the microstructure micrograph. With 20% addition of PALF, the crystal's length increased by 28.38 m (Figure 4f). Poor adhesion between the fibre and the gypsum crystals is the result of the fragmented sample's insufficient matrix and 100m wide pores. The inter transitional zone (ITZ) now has larger gaps as a result. The higher fibre count may have prevented the fibre from being encapsulated in the matrix during processing by obstructing matrix flow. As a result, the strength of the composites can be affected. Carvalho et al.[42] utilized SEM imaging to investigate how vacancies affect a pulp fibre composite. They found that gypsum crystal sizes varied across the fibre in samples with 10% pulp. They came to the conclusion that the fibre's form and hydrophilic qualities may be due to the larger size of the crystals around it. A strong medium for interlocking with the gypsum matrix is provided by the rough fibre surface. SEM measurements demonstrate that substantial fibre-matrix adhesion was achieved at a fibre volume fraction of 2%. With gypsum-cellulose composites, the same phenomenon has been verified in the research conducted by [49].The [50] looked into the morphological changes brought on by various NaOH concentrations on sisal-gypsum composites of 2 percent, 4 percent, 6 percent, 8 percent, and 10 percent weight. The findings shown that treated fibres outperformed untreated solutions in solutions containing 6% NaOH. The micrograph also reveals a fracture plane at the end of a sisal fiber at 20 percent volume, demonstrating the high fiber effect involved in the failure. This also corroborate with this present study when 20% PALF were added to the composite mixture, which affected the strength of the composite.




(a) C0 (b) C2

(c) C3 (d) C5

(e) C10 (f) C20

**Figure 4: Micrographs of 15mm PALF-gypsum Composites**

**B. Energy Dispersive X-ray Spectrometer (EDX)**

Figure 5 displays the EDX results of the control gypsum without fibre (C0) and the addition of 2% PALF (C2). Calcium, oxygen, and sulphur are the most common elements in the EDX charts for PALF-gypsum composites, whereas silicon and

carbon are minor contributors. This is in line with the research conducted by [51], in which Calcium, Oxygen and Sulphur were the chemical elements of gypsum present in both the interior of the cork and gypsum matrix. The specimen prepared with 2% PALF and a calcium value of 13.61 had no carbon, according to the EDX analysis. On the other hand, as the graph shows, the control sample had a greater carbon ratio (5.90). Gypsum's chemical components, calcium, sulphur, and oxygen (C-S-O), are present in both the gypsum matrix and the inside of PALF cells, proving that gypsum has entered these cells. Furthermore, at C2, the concentration of C-S-O increases from 94.11 percent to 100 percent. The results show that the wettability between the PALF and the gypsum paste is quite high at C2.

The results of the SEM pictures and mechanical characteristics are supported by the EDX images, which show that the gypsum with 2percent PALF added included more Ca, S, and O, which is the components of the calcium sulphate dihydrate crystal ($CaSO_4.2H_20$), as also supported by other researchers[6],[49]. The estimated component concentration is shown in Table 3.

**Table 3: EDX results of Plain Gypsum (CO) and Composite (C2)**

| Elements/ PALF/gypsum contents | O (%) | Ca (%) | S (%) | C (%) | Si (%) |
|---|---|---|---|---|---|
| C0 | 66.66 | 19.17 | 8.28 | 5.90 | - |
| C2 | 77.33 | 13.61 | 9.06 | 0.00 | - |
| C3 | 50.35 | 38.24 | 7.00 | 4.41 | - |
| C5 | 57.23 | 34.66 | 5.26 | 2.85 | - |
| C10 | 64.14 | 20.88 | 14.41 | 0.57 | - |
| C20 | 54.45 | 10.73 | 5.19 | 29.06 | 0.57 |

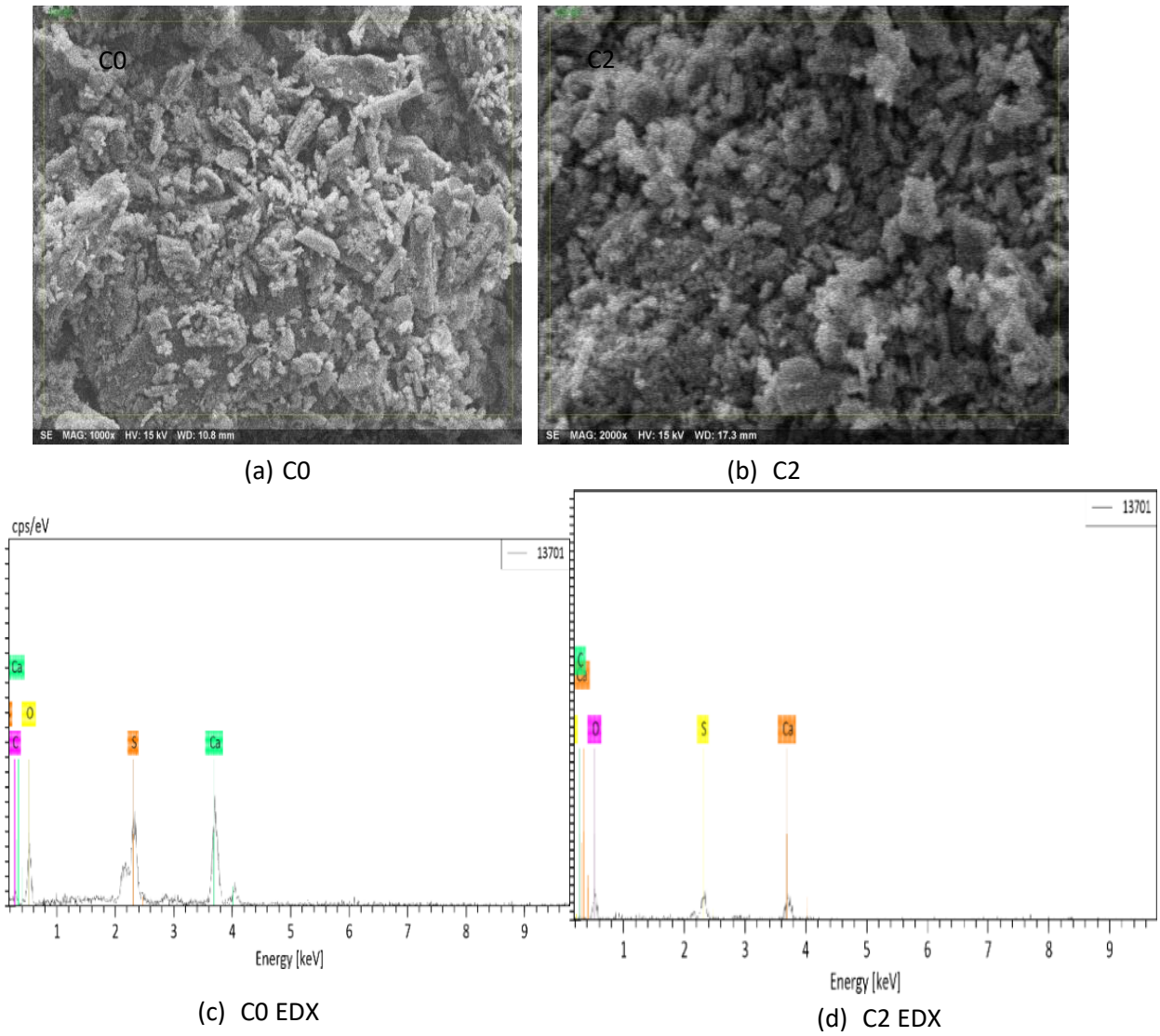
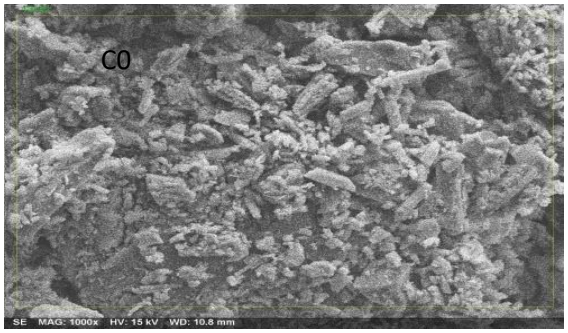
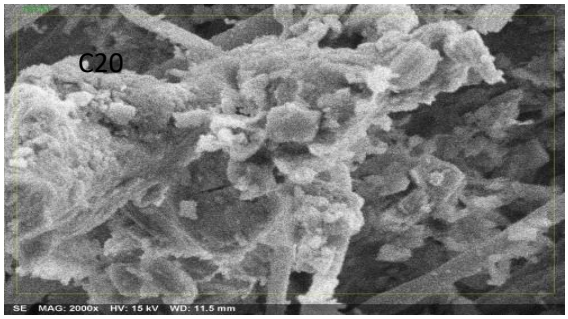



**Figure 5: EDX Spectra and Maps of Control Gypsum and C2**

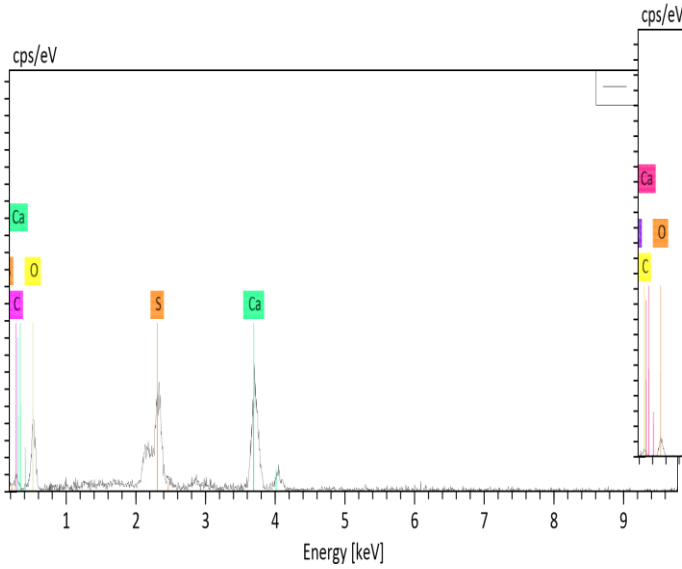
The presence of Ca, O, S, C, and Si in the C20 sample, as shown in Figure 6, points to the presence of expanded gypsum and ettringite minerals. At 19.17% and 10.73%, respectively, Figure 6 demonstrates that the Ca content in C20 is lower than that in the control specimen (C0). This may help to explain why C20 failed. The mechanical characteristics will be deteriorated if 20% PALF is added while containing the least amount of Ca and S. This is also in line with other research of gypsum based composite in which the mechanical properties was also reduced when greater percentages of fibre was added [52]



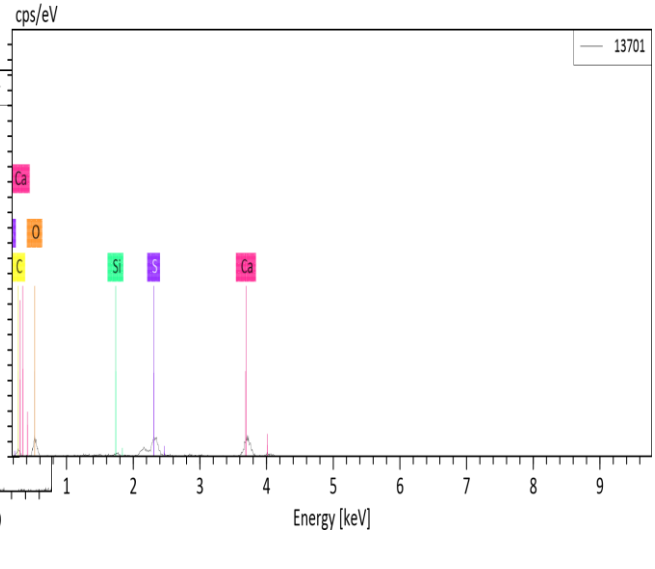

SEM for C0




(b) SEM for C20




(c) EDX analysis of C0




(d) EDX analysis of C20

**Figure 6: EDX spectra and maps of control gypsum and C20**

**C. X-Ray Diffraction Test of Gypsum PALF Composite**

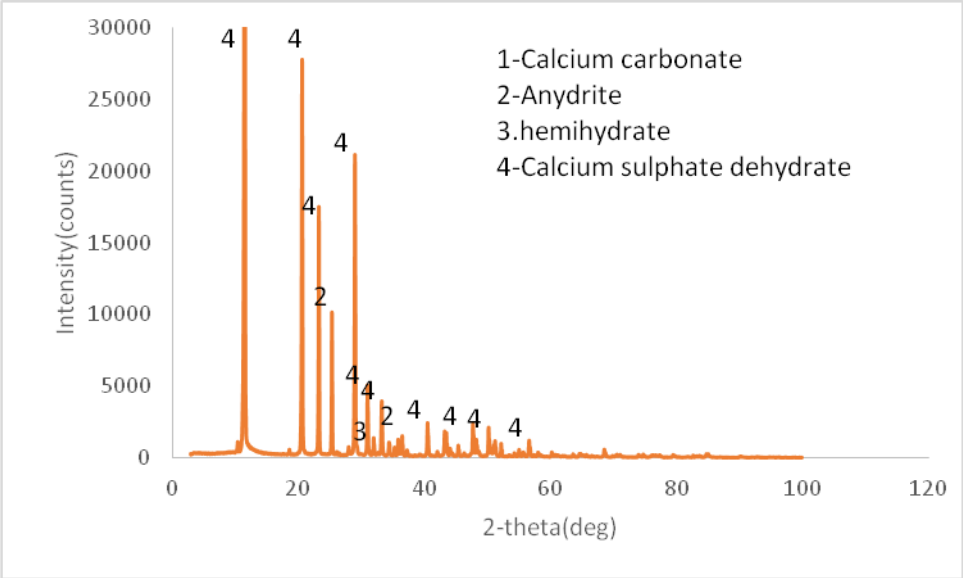
In this portion of the investigation, crystalline hydration products were qualitatively analyzed on a variety of samples using XRD analysis to spot any similarities and differences in the samples and to pinpoint the causes of the mixes' behavior. The hemihydrate transition is seen using diffraction patterns for b-hemihydrate (gypsum powder) (Figure 7a), gypsum dehydrate (Figure 7b), and PALF-gypsum composite (Figure 7c). Dihydrate gypsum ($CaSO_4.2H_20$) is the main crystal in the hydrated systems, with creation peaks appearing at 14.7, 20, 24, 33, 34, 40, 43, and 56, in a comparable way to the work conducted by [53], which is created as a result of the hydration of b-hemihydrate paste. The anhydrite peak is also seen in pure gypsum at 25.4 and 36.2. Anhydrite is produced during the production of hemihydrate, as evidenced by its presence in hemihydrate. All of the hemihydrate must have been moved to the dehydrated phase because there was no basanite phase ($CaSO_4.0.5H_20$) in the hydrated phase.

On the other hand, x-ray diffraction analysis of the PALF-gypsum composites shown in Figure 7c revealed that the primary crystalline component was small peaks of calcium sulphate dehydrate ($CaSO_4.2H_20$)

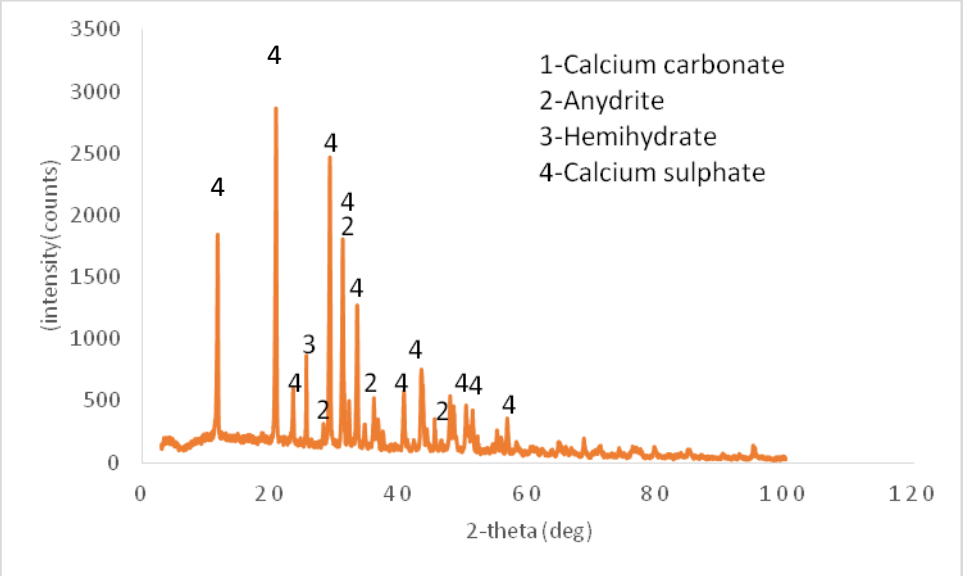
and calcium carbonate ($CaCO_3$). At 20, 28, 32, 44, and 56, there is a substantial amount of calcium carbonate. The variation in peak strength demonstrates that PALF has an impact on how well gypsum plaster hydrates (Figure 7c). It was discovered that adding PALF to the gypsum plaster reduced the hydration peak's intensity. The mechanical properties of composite materials were enhanced by calcite ($CaCO_3$). These findings are consistent with previous research [54]. Table 4 shows the study data and sample phases that were chosen at random from the literature.

**Table 4: Major phases found in sample material.**

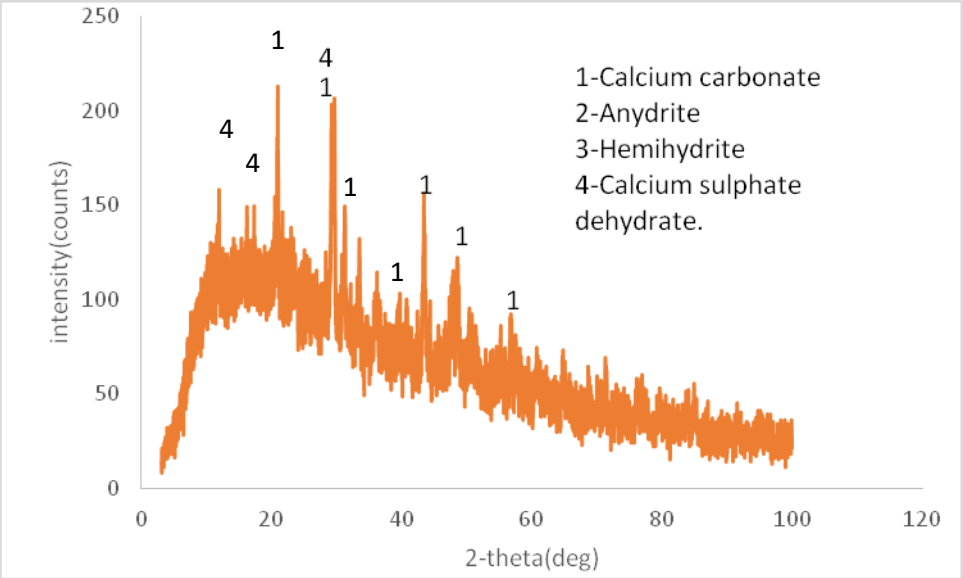
| Sample | Phases | Peaks at 2θ | Remarks |
|---|---|---|---|
| Hemihydrate | $CaSO_4.2H_2O$ | 14.6, 20, 24, 31.7, 34, 40, 49 and 56 | In the hydrated systems, the primary crystal and formation peaks of the dihydrate gypsum produced as a result of the hydration of b-hemihydrate paste can be seen. |
| | $CaSO_4$ | 38 | Pure gypsum has very few anhydrate peaks. |
| | $CaSO_4.0.5H_2O$ | 35 | This phase contained basanite with a lower intensity. |
| Hydrated hemihydrate | $CaSO_4.2H_2O$ | 11.6, 20.7, 24, 32, 34,40, 43 and 56 | This specimen has a more pronounced peak. |
| | $CaSO_4$ | 12, 14.7 and 29 | When water was introduced to the pure gypsum, this peak was raised. |
| Composite(PALF+GYPSUM) | $CaSO_4.2H_2O$ | 14.7 and 29.4 | Calcium sulphate dehydrate was detected as a small peak due to the presence of PALF. |
| | $CaCO_3$ | 20, 28, 32, 44 and 56 | Due to the presence of PALF, a modest peak of calcium sulphate dehydrate was found. |




(a) Gypsum hemihydrate




(b) Gypsum dihydrate




(c) PALF-gypsum composite.

**Figure 7: Gypsum hemihydrate, gypsum dehydrates, and PALF-gypsum composites' X-ray diffraction patterns**

## IV. Conclusion

In this study, the effects of various concentration of PALF on the microstructural characteristics of gypsum plaster were investigated. The following conclusions were drawn based on the microstructural properties experiments that were carried out.

The SEM results showed that at 2% fibre volume, a sticky surface coating formed around the gypsum crystals, which contributed to the high interlocking and compactness of the PALF-gypsum composite. It also demonstrates how composite materials experience considerable development as PALF percentage increases. The pores increase in number as percentages of PALF increases in the composites. The average pore size of these materials was 60 to 100 micrometres, as opposed to 27micrometers in the pure gypsum sample, according to SEM tests.

In contrast to when a higher amount of PALF was applied to the gypsum matrix, the between the gypsum crystal and 2% PALF was compact with a tiny gap. During the process of hydration, the structure change continuously as seen in X-ray diffraction diffractogram of the examined specimens which reveals a high peak of dihydrate gypsum. This peak was greatly lowered by the addition of PALF to the gypsum matrix. Different crystalline structures (needles, prism and fibres) were formed. The XRD diffractogram of the test specimen of the PALF-gypsum composite, which acts as a filler, showed a strong peak of calcium carbonate.

The findings of this experiment have conclusively shown that producing bio composite gypsum with a 2% PALF fraction is a viable option in enhancing their characteristics. The improvements in the performance of gypsum with 2percent

PALF addition suggests its broader application in construction industry for production of ceiling board, panels and partition boards.

**Acknowledgement**

The Universiti Teknologi Malaysia Research Management Centre (RMC), which provided assistance under Grant No. FRGS RJ130000.7822.4F887, is gratefully acknowledged by the authors. We greatly appreciate their assistance.

**Conflict of Interest**

The corresponding author says that all the other authors have no conflicts of interest.

**References**

[1] Dima C, Badanoiu A, Cirstea S, Nicoara A. I., and Stoleriu S., "Lightweight gypsum materials with potential use for thermal insulations," *Materials*, vol. 13, no. 23, 2020, pp. 1–13.

[2] Wirsching, F. Calcium Sulfate. *Ullmann's Encyclopedia of Industrial Chemistry*. https://doi.org/10.1002/14356007.a04_555, vol. A4, no. 44, 2000, pp 55-584.

[3] Awang Ngah S, Dams B, Ansell M. P, Stewart J, Hempstead R, and Ball R. J, "Structural performance of fibrous plaster. Part 1: Physical and mechanical properties of hessian and glass fibre reinforced gypsum composites," *Construltion Builing Materials*, vol. 259, no. 6, 2020, p. 120396.

[5] Gartner E. M, "Cohesion and expansion in polycrystalline solids formed by hydration reactions—the case of gypsum plasters," *Cement and Concrete Research*, vol. 39, no. 4, 2009, pp. 289–295.

[6] Yu Q. L and Brouwers H. J. H, "Microstructure and mechanical properties of β-hemihydrate produced gypsum: An insight from its hydration process," *Construction and Building Materials*, vol. 25, no. 7, 2011, pp. 3149–3157.

[7] Adrien J, Meille S, Tadier S, Maire E, and Sasaki L, "In-situ X-ray tomographic monitoring of gypsum plaster setting," *Cement and Concrete Research*, vol. 82, no. 2, 2016, pp. 107–116.

[8] Amathieu L and Boistelle R, "Crystallization kinetics of gypsum from dense suspension of hemihydrate in water," *Journal of Crystal Growth*, vol. 88, no. 2, 1988, pp. 183–192.

[9] Ridge M. J and Surkevicius H, "Hydration of calcium sulphate hemihydrate. I. Kinetics of the reaction," *Journal of Applied Chemistry*, vol. 12, no. 6, 1962, pp. 246–252,.

[10] Lewry A. J and Williamson J, "The setting of gypsum plaster - Part III The effect of additives and impurities," *Journal of Material Science*, vol. 29, no. 23, 1994, pp. 6085–6090.

[11] Lancia A, Musmarra D, and Prisciandaro M, "Measuring induction period for calcium sulfate dihydrate precipitation," *AIChE Journal*, vol. 45, no. 2, 1999, pp. 390–397.

[12] Solberg C and Hansen S, "Dissolution of CaSO4·1/2H2O and precipitation of CaSO4·2H2O - A kinetic study by synchrotron X-ray powder diffraction," *Cement and*

*Concrete Reserch*, vol. 31, no. 4, 2001, pp. 641–646.

[13] Gmouh A *et al.*, "Development and validation of a dimensional variation measurement set-application to the plaster setting," *Materials Science and Engineering, A*, vol. 372, no. 1–2, 2004, pp. 123–127.

[14] Karni, J and Karni, E. Gypsum in construction: origin and properties. *Materials and Structures*, vol. *28,* no. 2, 1995, pp. 92–100.

[15] Camarini G and De Milito J. A, "Gypsum hemihydrate–cement blends to improve renderings durability," *Construltion Builing Materials*, vol. 25, no. 11, 2011, pp. 4121–4125.

[16] Camarini G and Pinheiro S. M. M, "Microstructure of recycled gypsum plaster by SEM," *Advanced Materials Research*, vol.20, no.7, 2014

[17] Pinheiro S and Camarini G, "Characteristics of Gypsum Recycling in Different Cycles," *International Journal of Engineering and Technology*, vol. 7, no. 3, 2015, pp. 215–218.

[18] Skujans J, Vulans A, Iljins U, and Aboltins A, "Measurements of heat transfer of multi-layered wall construction with foam gypsum," *Applied Thermal Engineering*, vol. 27, no. 7, 2007, pp. 1219–1224.

[19] Gutiérrez-González S, Gadea J, Rodríguez A, Blanco-Varela M. T, and Calderón V, "Compatibility between gypsum and polyamide powder waste to produce lightweight plaster with enhanced thermal properties," *Construltion Builing Materials*, vol. 34, 2012, pp. 179–185.

[20] Toppi T and Mazzarella L, "Gypsum based composite materials with micro-encapsulated PCM: Experimental correlations for thermal properties estimation on the basis of the composition," *Energy Building*, vol. 57, no. 12, 2013, pp. 227–236.

[21] Gencel O, Del Coz Diaz J. J, Sutcu M, Koksal F, Álvarez Rabanal F. P, and Martínez-Barrera G, "A novel lightweight gypsum composite with diatomite and polypropylene fibers," *Construltion Builing Materials*, vol. 100, no. 113, 2016, pp. 732–740.

[22] Erbs A, Nagalli A, Querne de Carvalho K, Mymrin V, Passig F. H, and Mazer W, "Properties of recycled gypsum from gypsum plasterboards and commercial gypsum throughout recycling cycles," *Journal of Cleaner Production*, vol. 183, no. 2, 2018, pp. 1314–1322.

[23] Boccarusso L, Durante M, Iucolano F, Mocerino D, and Langella A, "Production of hemp-gypsum composites with enhanced flexural and impact resistance," *Construltion Builing Materials*, vol. 260, no. 2, 2020, p. 120476.

[24] Vimmrová A, Keppert M, Svoboda L, and Černý R, "Lightweight gypsum composites: Design strategies for multi-functionality," *Cement and Concrete Composites*, vol. 33, no. 1, 2011, pp. 84–89.

[25] Pedreño-Rojas M. A, Flores-Colen I, De Brito J, and Rodríguez-Liñán C, "Influence of the heating process on the use of gypsum wastes in plasters: Mechanical, thermal and environmental analysis," *Journal of Cleaner Production*, vol. 215, no. 09596526, 2019, pp. 444–457.

[26] Rubio-Avalos J. C *et al.*, "Flexural behavior and microstructure analysis of a gypsum-SBR composite material," *Material Letter*, vol. 59, no. 2–3, 2005, pp. 230–233.

[27] Bicer A and Kar F, "Thermal and mechanical properties of gypsum plaster mixed with expanded polystyrene and tragacanth," *Thermal Science and Engineering Progress*, vol. 1, 2017, no. 2, pp. 59–65.

[28] Vasconcelos G, Camões A, Fangueiro R, and Vila-chã N, "Gypsum-cork based composite material," *1st International Conference of Natural Fibers*, 1999, pp. 1–10.

[29] Deng Y. H and Furuno T, "Properties of gypsum particleboard reinforced with polypropylene fibers," *Journal of Wood Science*, vol. 47, no. 6, 2001, pp. 445–450.

[30] Ali M. A and Grimer F. J, "Mechanical properties of glass fibre-reinforced gypsum," *Journal of Material Science*, vol. 4, no. 5, 1969, pp. 389–395.

[31] Serhat Başpinar M and Kahraman E, "Modifications in the properties of gypsum construction element via addition of expanded macroporous silica granules," *Construltion Builing Materials*, vol. 25, no. 8, 2011, pp. 3327–3333.

[32] Khalil A. A, Tawfik A, Hegazy A. A, and El-Shahat M. F, "Effect of some waste additives on the physical and mechanical properties of gypsum plaster composites," *Construltion Builing Materials*, vol. 100, no. 68, 2014, pp. 580–586.

[33] Yakovlev G, Khozin V, Polyanskikh I, Keriene J, Gordina A, and Petrova T, "Utilization of blast furnace flue dust while modifying gypsum binders with carbon nanostructures," in *Environmental Engineering. Proceedings of the International Conference on Environmental Engineering*, 2014, vol. 9, p. 1.

[34] Yakovlev G, Polyanskikh I, Fedorova G, Gordina A, and Buryanov A, "Anhydrite and gypsum compositions modified with ultrafine man-made admixtures," *Procedia Engineering*, vol. 108, 2015, pp. 13–21.

[35] Khalil A. A, Tawfik A, Hegazy A. A, and El-Shahat M. F, "Influencia de distintas fuentes de sílice en las propiedades físicas y mecánicas de materiales derivados del yeso," *Materiales de Construccion*, vol. 63, no. 312, 2013, pp. 529–537.

[36] Bilici I, Deniz C. U, and Oz B, "Thermal and mechanical characterization of composite produced from recycled PE and flue gas desulfurization gypsum," *Journal of Composite Materials*, vol. 53, no. 23, 2019, pp. 3325–3333.

[37] Kuqo A and Mai C, "Mechanical properties of lightweight gypsum composites comprised of seagrass Posidonia oceanica and pine (Pinus sylvestris) wood fibers," *Construction and. Building Materials*, vol. 282, 2021, p. 122714.

[38] Eve S, Gomina M, Gmouh A, Samdi A, Moussa R, and Orange G, "Microstructural and mechanical behaviour of polyamide fibre-reinforced plaster composites," *Journal of the European Ceramic Society*, vol. 22, no. 13, 2002, pp. 2269–2275.

[39] Hernández-Olivares F, Bollati M. R,

Del Rio M, and Parga-Landa B, "Development of cork-gypsum composites for building applications," *Construction and Building Materials*, vol. 13, no. 4, 1999, pp. 179–186.

[40] Iucolano F, Liguori B, Aprea P, and Caputo D, "Thermo-mechanical behaviour of hemp fibers-reinforced gypsum plasters," *Construction and Building Materials*, vol. 185, 2018, pp. 256–263.

[41] Callister W. D and Rethwisch D. D, *Malzeme bilimi ve Mühendisliği*. Nobel, 2013.

[42] Carvalho M. A, Calil C, Savastano H, Tubino R, and Carvalho M. T, "Microstructure and mechanical properties of gypsum composites reinforced with recycled cellulose pulp," *Materials Research*, vol. 11, no. 4, 2008, pp. 391–397.

[43] Indian Bureau of Mines, "Indian Minerals Yearbook 2016 : Rare Earths," vol. 2016, no. 0712, 2018.pp. 245.

[44] Lei D. Y, Guo L. P, Sun W, ping Liu J, and wen Miao C, "Study on properties of untreated FGD gypsum-based high-strength building materials," *Construction and Building Materials*, vol. 153, no.6, 2017, pp. 765–773.

[45] Guan B, Yang L, Fu H, Kong B, Li T, and Yang L, "α-calcium sulfate hemihydrate preparation from FGD gypsum in recycling mixed salt solutions," *Chemical Engineering Journal*, vol. 174, no. 1, 2011, pp. 296–303.

[46] Huda M. S, Drzal L. T, Mohanty A. K, and Misra , "Effect of chemical modifications of the pineapple leaf fiber surfaces on the interfacial and mechanical properties of laminated biocomposites," *Composites Interfaces*, vol. 15, no. 2–3, 2008, pp. 169–191.

[47] Choy L. J, "Effects of extraction method on dry pulp yield and morphological properties of pineapple leaf fibre Dayangku Intan Munthoub , Wan Aizan Wan Abdul Rahman , Lew Jin Hau , Rohah A . Majid ," *Malaysian Journal of Fundamental and Applied Science*, vol. 16, no. 3, 2020, pp. 378–383.

[48] Li H, Xu C, Huang Y, Chen Q, Jiang Z, and Wang Y, "Modification on the Performance of the Hemihydrate Gypsum with the Plant Source Polymer of Dry Matcha Powder," *Journal Wuhan University of Technology, Materials Science Edition*, vol. 33, no. 6, 2018, pp. 1452–1458.

[49] Nindiyasari F*et al.*, "Characterization and mechanical properties investigation of the cellulose/gypsum composite," *Journal of Composite Materials*, vol. 50, no. 5, 2016, pp. 657–672.

[50] Alajmi R, "Study on sisal fibres as insulator in building materials," *Bachelor Thesis,University South. Queensland.*, 2015.

[51] Elkhessaimi Y, Tessier-Doyen N, and Smith A, "Effects of microstructure on acoustical insulation of gypsum boards," *Journal of Building Engineering*, vol. 14, no.4, 2017, pp. 24–31.

[52] Flores Medina N and Barbero-Barrera M, "Mechanical and physical enhancement of gypsum composites through a synergic work of polypropylene fiber and recycled isostatic graphite filler," *Construction and*

*Building Materials*, vol. 131, no. 100, 2017, pp. 165–177.

[53] Pundir A, Garg M, and Singh R, "Evaluation of properties of gypsum plaster-superplasticizer blends of improved performance," *Journal of Building Engineering*, vol. 4, no. 5, 2015, pp. 223–230.

[54] Middendorf B, "Physico-mechanical and microstructural characteristics of historic and restoration mortars based on gypsum: Current knowledge and perspective," *Geological Society Special Publication*, vol. 205, no. 1, 2002, pp. 165–176.