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Preparation and Characterization of Sound-Absorbent Based on Polystyrene Reinforced Primary Sludge and Fly Ash from Pulp Mill

Tengku Rachmi Hidayani[1], Basuki Wirjosentono[2*], Darwin Yunus Nasution[2], Tamrin[2]

[1]*Department of Agribisnis Kelapa Sawit, Politeknik Teknologi Kimia Industri, Medan, Indonesia, 20288*
[2]*Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Sumatera Utara, Medan, Indonesia, 20155*

Abstract. Efforts to utilize and improve the value of pulp waste have been done by using primary sludge (still containing cellulose) and fly ash as a filler to produce composite foam polystyrene (PS)-based as sound absorbent. In this study, the composite foam was produced by mixing primary sludge and fly ash with several compositions such as PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/3), (80/10/5/5), (75/10/10/5), and (75/10/12/3). The composite was characterized based on ISO 11654:1997, mechanical properties, thermal analysis using TGA and DSC, morphology using SEM, crystallinity using XRD, and chemical analysis using FTIR. The results showed that the band at 1600.63 cm^{-1} was sharper due to a chemical reaction being formed between PS, primary sludge, and fly ash. The XRD analysis showed a new diffraction peak in the $2\theta = 21\text{-}24^{o}$. The morphology analysis showed that primary sludge and fly ash were uniformly dispersed into PS, increasing the mechanical properties and decreasing the melting point of the PS foam composite. The sound-absorbing composite produced had qualified ISO 11654:1997 on the rating level of the sound absorption coefficient on materials for rooms with sound absorption classes D and C with the value of α_w 0.328-0.793.

Keywords: Fly ash; Polystyrene; Primary sludge; Pulp Mill; Sound-absorbing

1. Introduction

Noise is the pollution produced by some devices or tools which can affect human health by irritating the human nervous system. The main sources of noise exposure are vehicles, rail, metro, construction equipment, industrial enterprises, engineering equipment of buildings, and household noises. The noise is impossible to eliminate, but it can be reduced by using a sound absorbent (Nigmatullina *et al.*, 2020). Furthermore, the growth of industry, urbanization, and transportation increase noise pollution in the environment. Besides significantly affecting work efficiency and living standards, noise causes a series of health problems such as hearing loss, sleep disturbances, tiredness, and cardiovascular and psychophysiological problems (Yang *et al.*, 2020). Hence, controlling the noise from the living environment is important by using sound-absorbing materials.

Conventional sound-absorbent is normally delivered from metal, inorganic material, and organic polymer. However, the use of these materials affects the environment and human health negatively because of the manufacturing process which requires the use of

*Corresponding author's email: basuki@usu.ac.id,
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adhesives and foaming agents as chemical reagents. In addition, the materials are petrochemical products and the production consumes high energy (Issa, Mahmoud, and Hammad, 2021). Using natural fiber materials as sound absorbers has several benefits, such as less or no pre-treatment and modification in the manufacturing process, being environmentally friendly, easy availability, lightweight, low CO_2 emission, low price, and biodegradability (Yang *et al.*, 2020). For example, the low-cost sound insulation from textile waste and cork exhibits good sound-absorbing properties and can embed noise sources in certain frequency ranges 2800 Hz up. However, the effectiveness of the materials for sound-absorbent will represent a further stage of study (Luliana *et al.*, 2015).

Recycled porous material has comparable value to natural materials as a sound absorber. It also improves thermal insulation properties. A study reported that recycled materials from ethylene-vinyl acetate (EVA), polyvinyl chloride (PVC), polystyrene (PS), and polypropylene (PP), in different mixtures with a binding adhesive, can be considered as a good precursor of sound absorption suitable for room acoustic predictions (Biskupičová *et al.*, 2021). In addition, PS is one of the widely used plastic materials for packaging and insulation purposes. However, its waste generates hazards for humans, animals, and the environment. Composite-based PS is a good solution to deal with the waste of PS and can be used for building insulation due to its low-cost property and its ability to provide a long-term solution for Styrofoam waste (Abir, Faruk, and Arifuzzaman, 2020). PS is also one of the most common polymers which benefit from low thermal conductivity, high optical transmission, good heat stability, and excellent moisture resistance (Zhao *et al.*, 2021).

PS has been used as the most insulating material in Europe. The market share is about 80% due to its low cost and ease of processing (Heller and Flamme, 2020). PS and Portland cement as aggregate and binder were mixed as masonry in the construction. The result shows that the composite has efficient mechanical properties and meets the required parameters. In addition, compared to the commercial one, the composite was lighter, less permeable, and more economical (Hernández-Zaragoza *et al.*, 2013). It is also reported that sawdust reinforced with extended PS composites is superior to the synthetic fiber polymer as dumping sound material. Sound absorption of composite foam increased with increasing sawdust with loading level of 80% (Abdel-Hakim *et al.*, 2021). PS-fly ash composite is a potential construction material to solve the environmental problem by recycling waste fly ash and plastic material. It is reported that this composite material bears the low cost and water insulation. Therefore, it has the potential to be used as coating material on the walls and tiling in buildings (Bicer, 2020).

Fly ash, an agro-waste from any industry, is a second layer by-product from the combustion of fuel. It has been used as a precursor to producing nano-silica as filler for membranes, rubber airbags, composite, and concrete materials. The study reported that the presence of nano-silica improves the mechanical properties and durability of composite material (Indrasti *et al.*, 2020). While, it is stated that the relationship between the content of nano-silica and the mechanical properties of concrete needs further study to develop compressive strength that can be applied to any concrete mixture (Eddhie, 2017). Phosphate sludge is also used to prepare ceramic bricks by mixing with kaolin (Muliawan and Astutiningsih, 2018). Therefore, this study aims to utilize the by-products, fly ash, and primary sludge from the pulp industry as a filler to prepare PS composite foam as a sound-absorbing material. The pulp industry has been the subject of a long debate involving the local community due to the waste produced by factories. Therefore, this study is expected to be a solution for the utilization of waste. The new product is also expected to be a good formula to prepare a biodegradable damping material.

2. Methods

2.1. Materials

The materials used in this experiment were PS $(C_8H_8)_n$ type GPPS (General Purpose Polystyrene) injection grade, benzoyl peroxide ($C_{14}H_{10}O_4$), xylene $(CH_3)_2C_6H_4$, maleic anhydride ($C_4H_2O_3$), which were purchased from Merck (Darmstadt, Germany). Primary sludge and fly ash were obtained from Indah Kiat Pulp & Paper Tbk., Riau, Indonesia.

2.2. Preparation of Primary Sludge and Boiler Ash

Primary sludge was dried under the sun for 7 days and then dried in the vacuum oven at 100^o C for 6 hours. After drying, it was blended with a blender and sieved on a 400-mesh sieve. Meanwhile, fly ash was pulverized with a size of 200 mesh. It was purified using a 2 M HCl solution with fly ash: HCl ratio, 1:10, then distilled for 2 hours. The fly ash HCl solution was filtered and then washed using distilled water until pH-neutral. It was then calcinated at 600^o C for 2 hours to decompose and remove amorphous content. The calcinated fly ash was allowed to cool at room temperature and put into a ball mill (Planet PM 200) for 10 hours.

2.3. Preparation of Sound-Absorbing Polystyrene

PS was grafted using maleic anhydride as follows. The amount of 50 g of PS was weighed and then put into a reflux device. It was mixed with 100 mL of xylene solvent, 1% maleic anhydride, and 1% benzoyl peroxide and refluxed at 110^o C for 5 hours to produce PS-g-MA polyblend. The grafting procedure aimed to improve the chemical bonding between PS, primary sludge, and fly ash. Composite foam of PS-filled primary sludge and fly ash was carried out with a reflux reactor using xylene solvent at a temperature of 110^o C for 2 hours, with the help of maleic anhydride and benzoyl peroxide. The composite was molded by using a compression press. The composition of the samples (PS: PS-g-MA: primary sludge: fly ash) was (80: 10: 10: 0); (80: 10: 7: 3); (80: 10: 5: 5); (75: 10: 10: 5); and 75: 10: 12: 3). The scheme for PS composite foam preparation is presented in Figure 1.

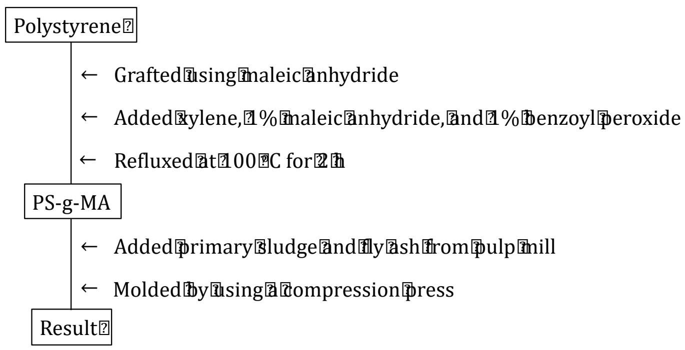

Figure 1 A flowchart of PS composite foam preparation

2.4. Characterization

The sound-absorbing composite of PS-reinforced primary sludge and fly ash was characterized using analytical techniques as follows. The morphology of the samples was investigated using scanning electron microscopy (SEM) Hitachi TM3030 (JEOL, Ltd., Tokyo, Japan) at an accelerating voltage of 10 kV. To reduce charging during analysis, the sample was first coated with a thin layer of gold. The chemical structure in the samples was analyzed using a Fourier-transform infrared (FTIR) spectrometer (Nicolet 380, Thermo Scientific, Boston, USA) in a transmission mode with a resolution of 2 cm^{-1} and 100 scans. The crystallinity of the samples was analyzed using X-ray diffraction (XRD) Bruker D8 advanced X-ray diffractometer (Bruker Optik GmbH, Ettlingen, Germany). The thermal properties of the samples were characterized using Thermogravimetric analysis (TGA), DTA/TG Exstar SII 7300 (Hitachi medical system, Tokyo, Japan) heated between 30 and 600° C with a heating rate of 10° C/min. It was also studied via a differential scanning calorimetry (DSC) X-DSC7000 (Hitachi medical system, Tokyo, Japan) in a range temperature from 30° to 600° C using a heating rate of 10° C/min. The mechanical properties of the sample were tested with a dead load hardness tester, shore A, at room temperature according to ASTM D 2240. Finally, the sound absorption of the samples was carried out using an impedance tube according to ASTM E-1050-98. The amplitude from the graph was obtained by looking at the maximum value of each channel, and the sound absorption coefficient value was calculated using MATLAB software.

3. Results and Discussion

3.1. FTIR and X-ray Analysis

FTIR spectra aim to study the functional groups, chemical structures, and chemical compositions contained in fly ash, primary sludge, and PS-composite foam. FTIR spectra of the samples are presented in Figure 2.

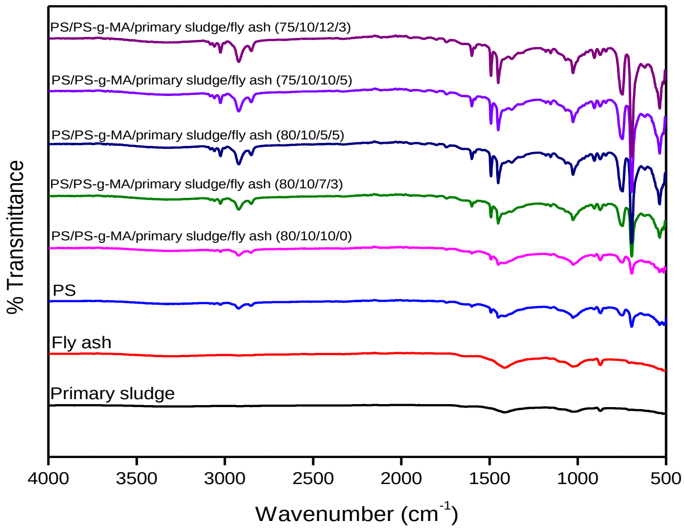

Figure 2 FTIR spectra of primary sludge, fly ash, PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5), (80/10/5/5), (75/10/10/5, and (75/10/12/3)

The peak at the band 3000–3500 cm^{-1} indicates the OH stretching vibrations in all samples (Kusrini *et al.*, 2021). In the sludge sample, the OH wave number was at 3744.40 cm^{-1} with a fairly wide peak. This is due to the large amount of water in the sample. The band at 2850.31 cm^{-1} attributed to H-C-H stretching (alkyl, aliphatic), 1623.65 cm^{-1} corresponded to OH-fibers (water absorption), and 1414.47 cm^{-1} was vibrations of HCH and OCH bonds (methyl groups). In the primary sludge, there was a band at 706.52 cm^{-1} indicating 1β crystalline cellulose polymorph from the pulp (Risnasari *et al.*, 2018).

For PS, there was a peak at 1492.25 cm^{-1} indicating the absorption of the indentation of the (C-H) group as alkyl. The peak at 1600.63 cm^{-1} is attributed to the bond in benzene with strong to weak intensity. The band at 2922.36 cm^{-1} and 3059.46 cm^{-1} corresponded to C-H bonds with benzene and Ar-H. In the fly ash, it can be seen that the absorption at 1621.90 cm^{-1} presented C=O stretching on the ketone. The peak of 1064.25 cm^{-1} and 796.10 cm^{-1} attributed to C=C stretching of hemicellulose and C=C stretching of vinylidene alkenes, respectively. The aromatic ring in fly ash was seen at the band 777.60 cm^{-1} (Reza *et al.*, 2020; Huan *et al.*, 2015).

Furthermore, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5), (80/10/5/5), (75/10/10/5), and (75/10/12/3) showed a decrease in the peak in the absorption area of 3059.46 cm^{-1}. The band at 1600.63 cm^{-1} also looked sharper. From the FTIR results, it can be concluded that PS, primary sludge, and fly ash have been physically blended, and there were chemical reactions formed during the mixing process as reported by previous studies (Oromiehie, Ebadi-Dehaghani, and Mirbagheri, 2014).

XRD patterns of PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) and (75/10/10/5) are plotted in Figure 3. Concerning Figure 3, after the blending of PS with PS-g-MA, primary sludge, and fly ash, it was observed that there was a new diffraction peak in the $2\theta = 21 - 24^{o}$ corresponding to the crystalline region. The increase in the crystallite value for the sound-absorbing composite foam PS/PS-g-MA/primary sludge/fly ash contributed to the mechanical properties of the resulting sound-absorbing composite foam.

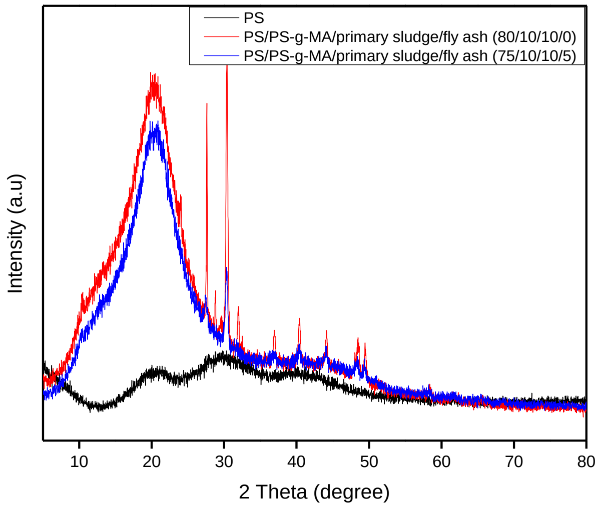

Figure 3 XRD pattern of PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) and (75/10/10/5)

3.2. Morphological Characterization

Figure 4 shows the morphology of primary sludge, fly ash, and PS/PS-g-MA/primary sludge/fly ash (75/10/10/5) composite. Figure 4(a) shows that primary sludge had a flat and rough surface with a few bumps on the surface of the sample. This is due to the primary sludge obtained directly from pulp mill waste being manually processed. While fly ash in Figure 4(b) shows a rough surface and there were longitudinal tears. In this study, the boiler ash obtained was calcined at a temperature of 600^{o} C which was converted into bio-solid (coal), bio-liquid (bio-oil), and biogas during the heating. At high temperatures, C-H and C-O bonds were breakdowns due to the continuous char devolution (Younis *et al.*, 2018). In addition, PS, as shown in Figure 4(c) had a flatter and smoother surface compared to primary sludge and fly ash. Figure 4(d) shows the surface morphology of PS/PS-g-MA/primary sludge/fly ash (75/10/10/5) after the addition of maleic anhydride, primary sludge, and fly ash containing several pores with relatively small sizes. However, from the observations, it could still be concluded that the filler was well distributed and dispersed into the polymer matrix and no agglomeration was produced.

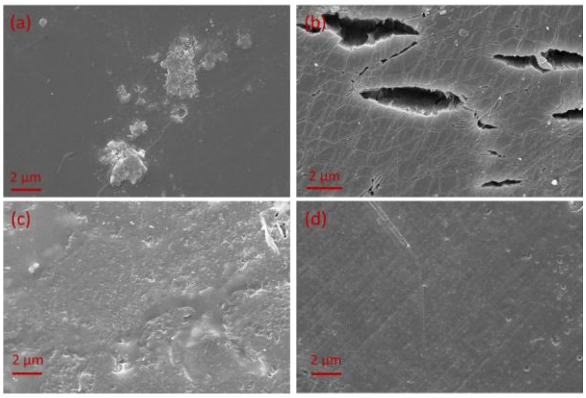

Figure 4 SEM micrograph of (a) primary sludge, (b) fly ash, (c) PS, and (d) PS/PS-g-MA/primary sludge/fly ash (75/10/10/5) with magnification 3.5 kX and scale bar 2 µm

3.3. Thermal Analysis

The thermal properties of the sample in this study were characterized using TGA and DSC. The TGA curve for all samples is presented in Figure 5, and the DSC thermogram for PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) and (75/10/10/5) is shown in Figure 6.

Figure 5 shows that all samples had three main distinct degradations. The first step was at 100° C with a mass loss of around 2% for PS, and almost 10% for PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5), (80/10/5/5), (75/10/10/5), and (75/10/12/3). The second step was from 200 to 350 °C for PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5), (80/10/5/5), (75/10/10/5). However, PS/PS-g-MA/primary sludge/fly ash (75/10/12/3) experienced a lower temperature between 131 and 371° C. At this step, all samples lost about 20% of their mass. Finally, PS decomposed at 349-434° C with a residual mass of 0,6%.

Figure 6 shows that PS had a glass transition (Tg) of 92° C and melting point (T_m) of 430.79° C. With the addition of primary sludge and fly ash onto PS, it can be seen that the T_m of the sound-absorbing composite foam for PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) and (75/10/10/5) decreased to 395.06° C and 401.00° C, respectively. This is probably due to physical interactions between PS, PS-g-MA, primary sludge, and fly ash as described in the FTIR data. In addition, the presence of a filer in the material can reduce the melting point of the sound-absorbing composite foam (Hidayani *et al.*, 2021).

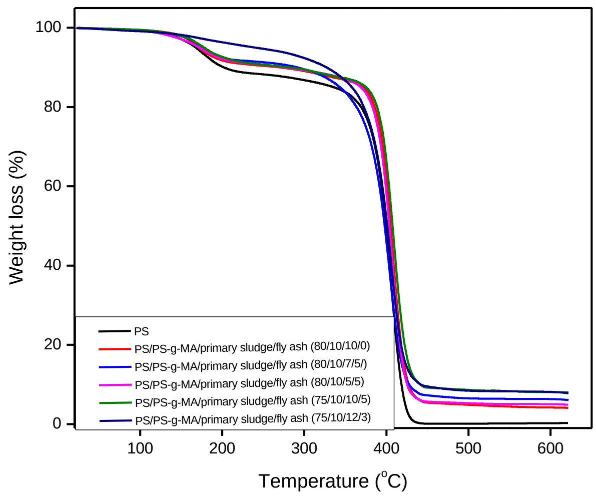

Figure 5 TGA curve of PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5/), (80/10/5/5), (75/10/10/5), and (75/10/12/3)

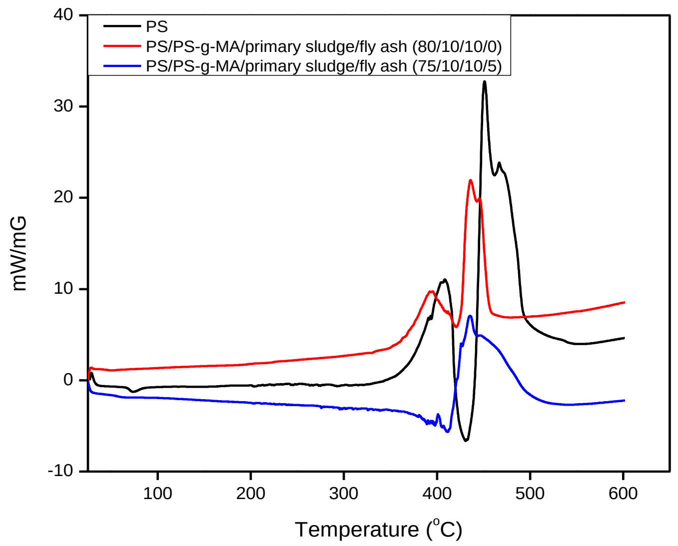

Figure 6 DCS thermogram for PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) and (75/10/10/5)

3.4. Mechanical Properties

Figure 7 shows the strain and stress curves of all samples. In general, the tensile strength of a composite foam significantly increased after the addition of filler. PS and PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) had a tensile of 6.526 MPa and 5.841 MPa, respectively. PS/PS-g-MA/primary sludge/fly ash (80/10/7/3) had a tensile of 6.043 MPa which was the highest value compared to all samples.

The increase in tensile strength after the addition of primary sludge/ash with composition 7/3 was because the filler was well and uniformly dispersed into the matrix. The addition of filler increased the tensile strength and Young's modulus indicating that sufficient dispersion of the filler into the matrix has occurred. In addition, as the filler concentration increased, the sample deformation decreased. A previous study reported that the wall panel with the presence of sludge increased the tensile strength by 0.61 N/mm^2 compared to without sludge, 0.59 N/mm^2 (Hidayani *et al.*, 2021).

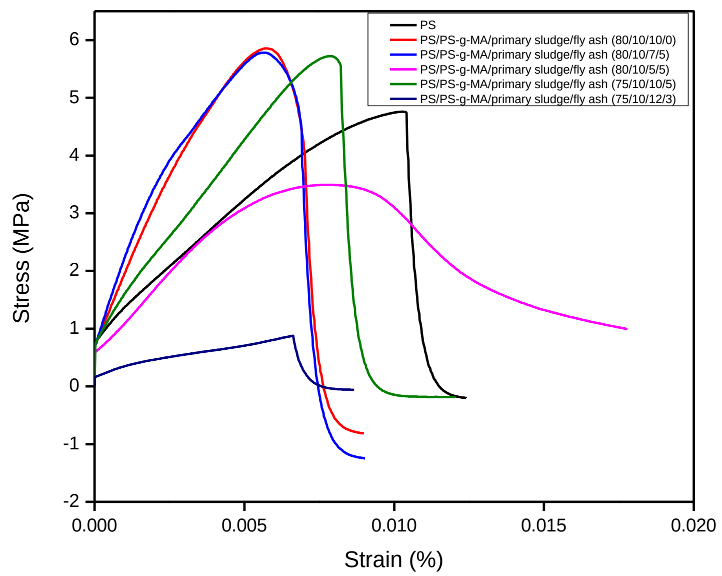

Figure 7 Stress-strain curve for PS, PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), (80/10/7/5), (80/10/5/5), (75/10/10/5), and (75/10/12/3)

3.5. Sound Absorption Properties

The sound absorption coefficient of composite foam tested with a frequency between 200 and 2000 Hz is shown in Figure 8.

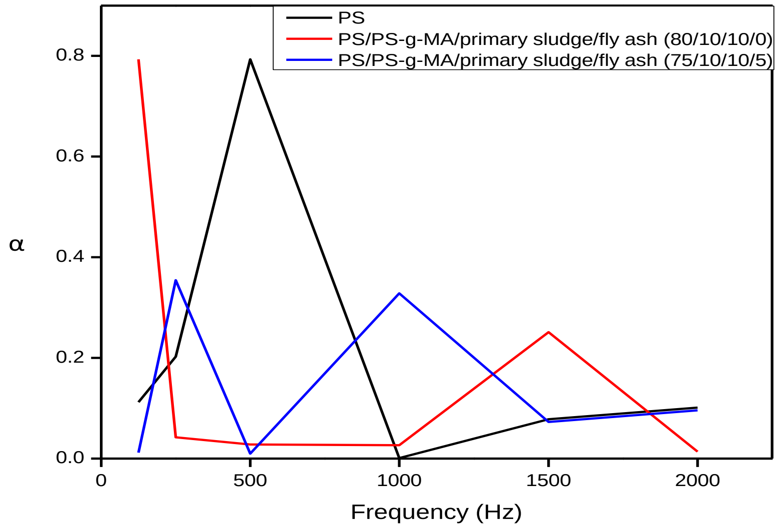

Figure 8 The sound absorption coefficient of the composites

Regarding Figure 8, it can be seen that the absorption coefficient of PS/PS-g-MA/primary sludge/fly ash (80/10/10/0) decreased from a frequency of 200 Hz to 400 Hz. On the other hand, PS and PS/PS-g-MA/primary sludge/fly ash (75/10/10/5) increased in the absorption coefficient at the range frequency. The highest value for PS was 0.793 at a frequency of 500 Hz and then experienced a dramatic decrease. Although the absorption coefficient of PS/PS-g-MA/primary sludge/fly ash (75/10/10/5) had decreased at a frequency of 500 Hz, it increased again and had the highest peak at a frequency of 1000 Hz with a value of 0.328. For PS/PS-g-MA/primary sludge/fly ash (80/10/10/0), the absorption coefficient continued to decrease. The sound-absorbing composites produced have qualified ISO 11654:1997 about the rating level of the sound absorption coefficient on materials for rooms with sound absorption classes D and C with the value of α_w 0.328-0.793.

4. Conclusions

Sound-absorbing composite PS-based filled with primary sludge and fly ash from a pulp mill has been produced by using a compression press. From the results, primary sludge and fly ash are dispersed uniformly onto the PS polymer matrix which increases the mechanical properties. It also shows that chemical bonds formed during the mixing process due to the grafting procedure between PS and maleic anhydride. The sound-absorbing composite has the value of α_w 0.328-0.793 which is qualified based on ISO 11654:1997 and sufficient for sound absorption for rooms. The composite produced is potentially used as a green and biodegradable sound absorber material. For future work, fly ash will be modified into nano-silica so that the mechanical properties of the composite increase and bear the parameter of the commercial damping sound.

Acknowledgments

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