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Designing 3D Printed Electromagnetic Wave Absorber

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ABSTRACT

Recent years, electromagnetic (EM) wave radiation has been regarded as an alarming danger for commercial appliances, biological systems, and high-quality information technology. The fabrication of EM wave absorbers is a crucial technological advancement in the field of EM wave management that can absorb the EM wave interference. Various materials that were developed to absorb EM wave interference are high cost, environmental impact, and are harmful to human health and other equipment. Therefore, this research introduces single and multilayer absorber that were fabricated by using the filler materials based on silica from beach sand and mill scale from industrial waste. The prepared polymer composite samples were also 3D printed with the desired shape and size so that it met the requirement of using it for any applications. The single layer and multilayer polymer composite thickness were fixed at 1 mm and 2 mm respectively. The microstructural phase formation, morphological study, particle size analysis and elemental analysis were analyzed by using X-ray diffractometer (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and Energy Dispersive X-ray (EDX) respectively. The microwave characterization of the polymer composite samples was analyzed by using Vector Network Analyzer (VNA) at 4 to 8 GHz frequency range. The results shows that single layer and multilayer have triple resonance peaks and may absorb the electromagnetic wave with the minimum reflection loss peak in between 5.5 to 6.5 GHz. It shows that the sustainability factor and low cost of silica-based sand and industrial waste-mill scale makes it highly attractive as a potential filler material that can be used as an EM wave absorber in many applications.

1. Introduction
1.1 Research Background

The problem of electromagnetic (EM) interference to communication devices poses significant challenges in maintaining reliable and efficient communication systems. EM interference can arise from various sources, including nearby electronic devices, power lines, radio frequency interference,

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and other wireless communication signals. This interference can disrupt communication signals, degrade signal quality, and lead to communication failures, impacting critical operations and causing inconvenience in everyday life and are harmful to human health. Kuila *et al.,* [1] reported that by reducing EM wave pollution generated by electronic devices and power lines, these absorbers contribute to a healthier electromagnetic environment for both humans and ecosystems. This will help preserve biodiversity and ensure the well-being of living organisms. The fabrication of EM wave absorbers holds immense potential for various industries and everyday life. Advanced fabrication techniques can significantly improve wireless communication systems, radar technology, healthcare diagnostics, and environmental sustainability. The benefits extend to improved connectivity, national security, enhanced medical imaging, and a healthier electromagnetic environment, benefiting intended users and society.

Silica is one of the most abundant substances that can be found in a variety of natural minerals including clay, rice husk, palm shells and silica sand. However, the highest silica content can be found in silica sand that can approach to 99% as reported by several authors [2-6]. Therefore, silica sand is a suitable material to produce low cost and eco-friendly EM wave absorbing material based on certain characteristics of silica sand. Silica sand can be classified as a sustainable natural resource as it is a commonly found mineral in the Earth's crust, typically in the form of quartz. Silica sand is also considered a rapidly renewable resource as the current usage rate of silica sand by mankind is inferior to the amount of silica sand that is generated naturally. The Department of Mineral and Geoscience Malaysia (JMG) have estimated that approximately 148.4 million tonnes of silica sand reserves located cumulatively in the states of Johor, Perak, Terengganu, Kedah, Kelantan, Sabah, and Sarawak.

Furthermore, ground silica acts as a functional extender to add durability, anti-corrosion and weathering properties in epoxy-based compounds, sealants, and caulk. Additionally, the procurement of silica sand is an easy and low-cost process compared to other materials used for EM wave absorbing material. Emerging technologies also allow the usage of silica sand sourced from natural waste composites that provide an eco-friendly aspect to the material. Recent reports by Wei *et al.,* [7] show that beach sand containing silica has a high reflection loss of -11.74 dB at frequency 12.4 GHz with a narrow bandwidth of 0.4 GHz makes it a strong candidate to replace carbon nanofiber (CNFs) as an EM wave absorbing material. Moreover, silica-based sand is lightweight and has an effective absorption bandwidth for two- and three-layer composite absorbers as reported by Liu *et al.,* [8]. It is also expected to perform better when used as a filler for Electromagnetic Wave (EMW) absorption than other materials like electronic waste, barium ferrite, carbon nanotube, and manganese zinc ferrite.

Mill scale is a by-product from the steel industry that currently poses a significant environmental hazard. Mill scale consists of the mixed iron oxides; iron (II) oxide (FeO), iron (III) oxide (Fe_2O_3) and iron (II, III) oxide (Fe_3O_4, magnetite) are commonly employed as an iron precursor. Idris *et al.,* [9] reported that mill scale has been utilized by making the nanoparticles mill scale as catalyst in synthesizing carbon nanotubes (CNTs) to be used as EM wave absorber filler. Other than that, Magnetite (Fe_3O_4) and Cobalt ferrite ($CoFe_2O_4$) were also used as catalyst to grow CNTs as reported by Idris *et al.,* [10]. Despite having significant absorption capacity, conventional absorbing materials like ferrite and magnetic absorbers have a high density as reported by Ashraf *et al.,* [11]. Furthermore, the permeability sharply decreases with increasing frequency.

Various materials have been developed as EMW absorption and focused on high performance. However, the materials present significant challenges such as high costs, negative environmental impacts, potential health risks and limiting their broader application. Hence, the novelty and uniqueness of this research work is highlighted by introducing silica beach sand and industrial waste mill scale as low-cost filler materials to be further 3D printed and used as an EM wave absorbing material. Thus, this research work creates cost-effectiveness and environmentally friendly. It also provides practical solution to the EMW absorption problem that focuses on sustainability, low-cost and better performance at wide frequency range potential for various applications. This research also addresses some of the challenges in EM wave absorbing applications. The investigation will be crucial in developing a new generation EM wave absorbing material manufactured from natural sources and industrial steel waste.

1.2 Significance of the Research

Previous research has been done on a variety of materials to be utilized as an EM wave absorber (EMWA), and the research did succeed in producing a greater absorption of EM waves. However, a thicker sample or a higher filler content are used in the research project. Therefore, this research work synthesizes and investigates the EM wave absorption properties on 3D printed EMWA that have not been studied in prior research. In this study, the EMWA was made by developing lightweight and thin multilayer 3D printed EM wave absorbers from natural sources and industrial waste to reduce electromagnetic wave interference in advanced radar technology. Additionally, this study also presents a low-cost mill scale from industrial waste. The single and multilayer 3D printed EM wave absorber design based on natural sources and industrial waste have good performance in absorbing the EM wave compared to other high-cost materials.

The characteristics of the synthesized 3D printed polymer composite being produced are lightweight, thin thickness and low-cost by using silica-based beach sand and industrial mill scale waste being converted into valuable product. By using natural sources and industrial waste, it gives significant benefit to electromagnetic absorbers since they lie in their potential for promoting environmental sustainability. It may also reduce the electromagnetic interference pollution by absorbing the unwanted wave or signal. Moreover, the prepared 3D printed prototype may be designed with different size and shape according to the application needed.

2. Methodology

2.1 Preparation of Filler Materials

The process starts with the collection of raw materials–silica sand and mill scale. Silica is a naturally occurring mineral composed of silicon dioxide (SiO_2) taken from the beach sand, and mill scale is a byproduct of steel manufacturing. The collected raw materials were crushed into powder form and washed to remove any impurities and dirt that may be present on their surfaces. This washing was typically done using a water bath, where the materials were immersed in water and agitated to dislodge impurities. After washing, the materials were filtered to separate the cleaned material from the water and impurities. Following the washing and filtration, the wet materials were dried in an oven at 100° C for 1 hour as shown in Figure 1. Drying is essential to remove any remaining moisture, ensuring that the materials are in a dry state for further processing.

To acquire smaller and homogeneous particle size, the dry sand was crushed using High Energy Crusher with range of spindle speed (r.p.m) between 26 000 to 28 000 r.p.m. The dry silica and mill scale were then sieved through a 100 µm stainless steel sieve as shown in Figure 2. Sieving helps to achieve a consistent and finer particle size. The sieve acts as a filter to separate particles larger than 100 µm from those that are smaller. The resulting fine particles are desirable for uniform dispersion in the polymer matrix.

(a) (b) (c)

Fig. 1. (a) Wet mill scale (b) Oven for curing sample (c) Cured mill scale

(a) (b) (c)

Fig. 2. (a) Dry silica powder (b) crushed silica powder (c) sieved powder

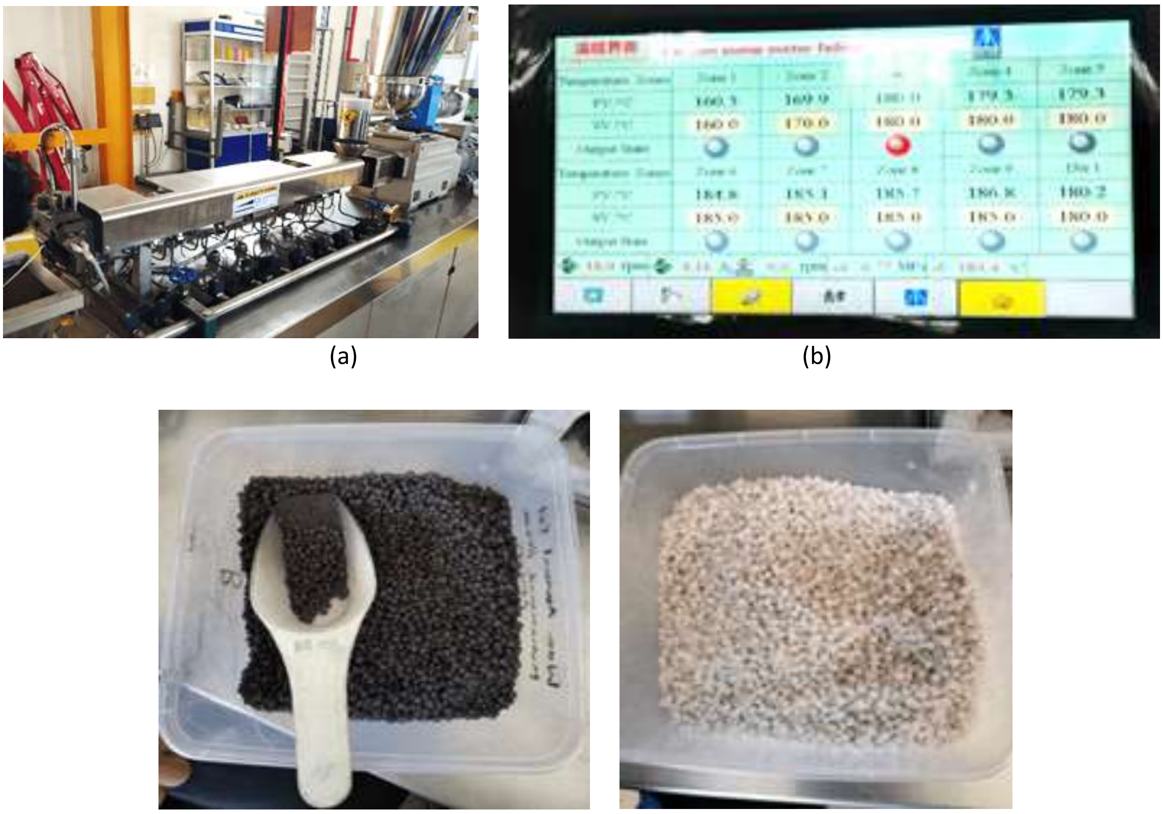
The sieved silica was subjected to sintering at around 1100°C for a duration of 6 hours in a furnace as shown in Figure 3 to obtain the complete phase of SiO_2. Sintering is a process in which the powdered material is heated to a temperature below its melting point, causing particles to fuse together. This step can enhance the mechanical properties of the material.

(a) (b) (c) (d)

Fig. 3. (a) Furnace (b) Ceramic boat located in the furnace heating zone (c) Before sinter (d) Sintered silica powder

2.2 Preparation of Filament

The sintered silica and mill scale were prepared as filler materials. Filler materials were added to a polymer matrix to enhance certain properties and characteristics. In this case, 5wt% of filler materials were added to the polylactic acid (PLA) matrix. The composite material, consisting of the filler materials and PLA, was then processed through a Twin-Screw Extruder (TSE) as shown in Figure 4. This extrusion process involves heating the composite and forcing it through a die to produce filament with a consistent diameter. The temperature during extrusion was controlled between 160°C to 185°C to ensure proper melting and blending of the PLA and filler materials as shown in Table 1.



(a) (b)

(c) (d)

Fig. 4. (a) Twin screw extruder (b) Setup of heating temperature (c) Mixture of mill scale and PLA pellet and (d) mixture of silica and PLA pellet

Table 1
Setup of heating temperature

Zones	Temperature, ^{0}C
1	160
2	170
3	180
4	180
5	180
6	185
7	185
8	185
9	185
Die 1	180

The resulting filament with a diameter of 1.75 mm was used as feedstock for a 3D printer, the FlashForge Creator Pro 2 as shown in Figure 5. 3D printing allows us to create physical objects layer by layer based on a digital model. The design for 3D printing was done using TinkerCAD software. The thickness of 3D printed single layer and multilayer sample were fixed to 1 mm and 2 mm respectively.

(a) (b)

Fig. 5. (a) 3D printer (FlashForge Creator Pro 2) (b) 3D printed sample

Marpaung *et al.,* [12] reported that for a double-layer absorber composed of the layer 1 (Silica) and layer 2 (Barium hexaferrite) as the absorption layer and matching layer respectively. Reflection loss (RL) of double-layer can be calculated and simulated using the transmit line theory by the following Eqs. (1) to (3) as also reported by several authors [13-15].

$$RL(dB) = 20\ log\left[\frac{Z_2 - 1}{Z_2 + 1}\right] \tag{1}$$

$$Z_1 = \sqrt{\frac{\mu_1}{\varepsilon_1}}\ \tanh[\ j(\frac{2\pi f d_1}{c})\sqrt{\mu_1 \varepsilon_1}\ ] \tag{2}$$

$$Z_2 = \frac{\sqrt{\frac{\mu_2}{\varepsilon_2}}\,(\ Z_1 + \sqrt{\frac{\mu_2}{\varepsilon_2}} \tanh[\ j(\frac{2\pi f d_2}{c})\sqrt{\mu_2 \varepsilon_2}\ ]}{\sqrt{\frac{\mu_2}{\varepsilon_2}} + Z_1 \tanh[\ j(\frac{2\pi f d_2}{c})\sqrt{\mu_2 \varepsilon_2}\ ]} \tag{3}$$

Where, μ_1 and μ_2 are complex permeability ($\mu = \mu' - j\mu''$) and ε_1 and ε_2 are complex permittivity ($\varepsilon = \varepsilon' - j\varepsilon''$), *f* is frequency. d_1 and d_2 are the thickness of absorbing layer and matching layer, respectively. *c* is the velocity of light.

2.3 Materials' Characterization

X-Ray Diffraction Analysis (XRD) was employed to determine the crystalline structure and phase composition of the material, helping to identify different crystallographic phases. On the other hand, Transmission Electron Microscope (TEM) was used to obtain the particle size analysis. Field Emission Scanning Electron Microscope (FeSEM) was used to closely examine the surface morphological and microstructural properties of the sample. The elemental analysis was further measured by using Energy Dispersive X-ray (EDX). After 3D printing process, the fabricated polymer composite was subjected to measurements by using Vector Network Analyzer (VNA) to measures the electromagnetic performance of the sample at frequency C-band (4-8 GHz) and to assess its ability of absorbing the electromagnetic radiation.

3. Results and Discussion

3.1 Phase Analysis

X-ray diffraction pattern of sintered silica and mill scale is shown in Figure 6. The X-ray diffraction patterns was compared with the standard reference code data. The phase obtained for mill scale are magnetite (Fe_3O_4) and wustite (FeO) as compared to reference code data for Fe_3O_4 (01-089-3854) and FeO (01-074-1886). There were eight peaks presence in mill scale and the peak was sharp showing crystalline phase and larger particle size. On the other hand, the phase observed for silica is single phase of crystalline silica (SiO_2) as also reported by Rampe *et al.,* [16]. The presence of peaks at certain angle showing the crystal structure presence in the sample. According to several authors [17,18], silica is crystalline and have various crystalline forms. Therefore, from the X-ray diffraction patterns, it can be observed that the single phase of silica have the highest diffraction peak at an angle 2θ of ~22^0. This result is match with the EDX results that shows the SiO_2 is dominant in the sample.

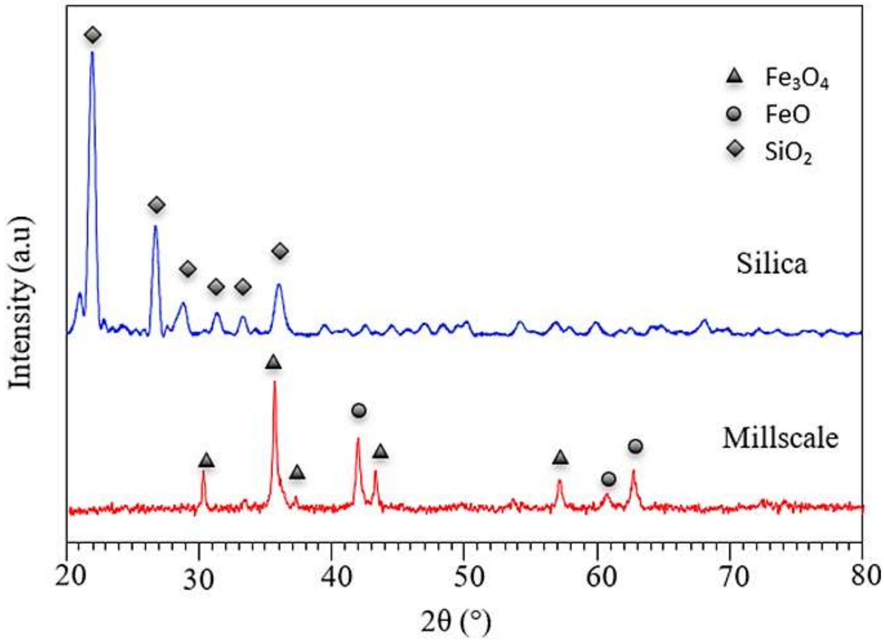


Fig. 6. X-ray diffraction pattern of sintered silica and mill scale

3.2 Particle Size Analysis

The particle size analysis obtained using TEM as shown in Figure 7 showing the particles are slightly agglomerated although the powder was dispersed in an ethanol solution for 30 minutes. The highest percentage for the range of particle size distribution is in between 200 to 400 nm. However, the average particle size of mill scale is 430.32 nm.

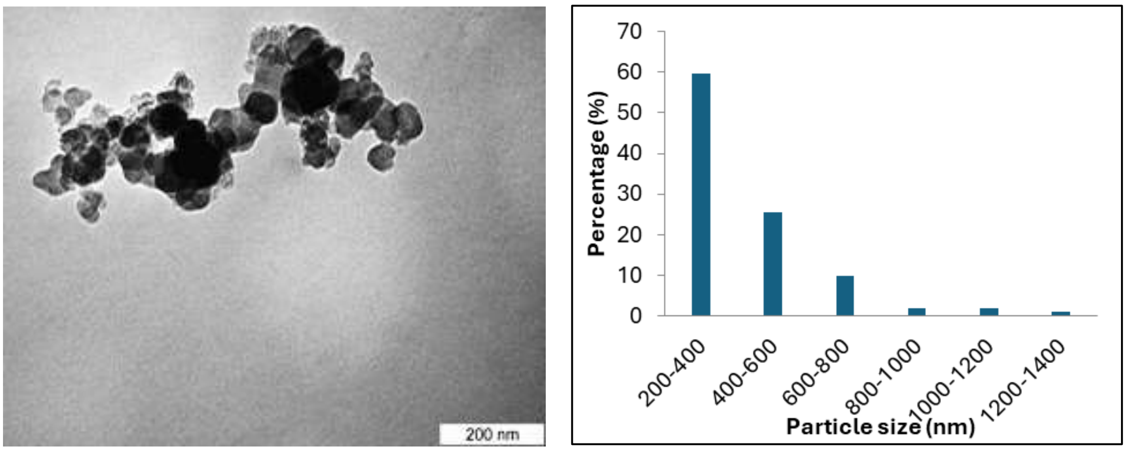
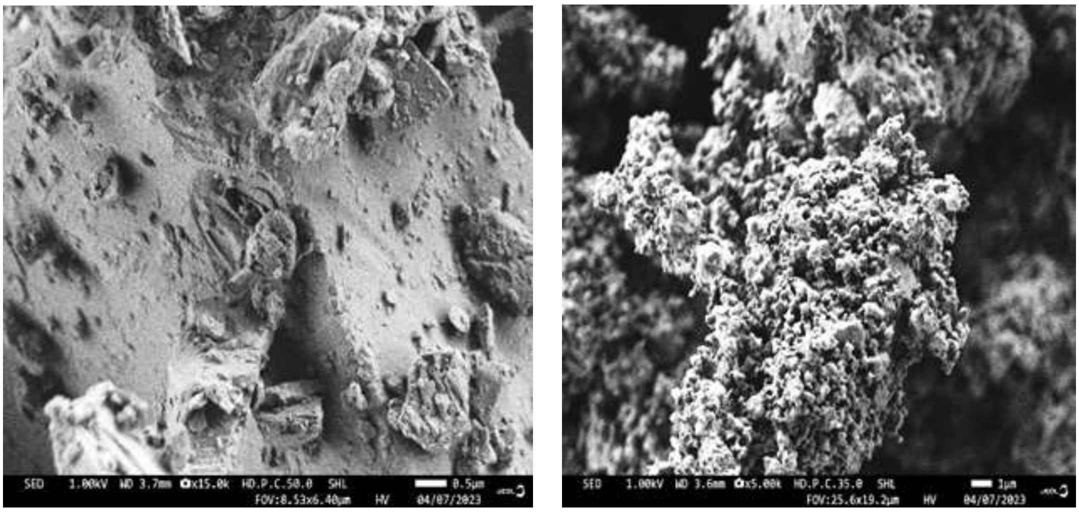


Fig. 7. TEM picture and corresponding particle size histogram of mill scale

3.3 Microstructural Analysis

Figure 8 shows the FESEM image of raw silica sand and sintered silica at magnification of 15k and 5k respectively. The morphology of the raw silica particles is in irregular shape. The sintered silica morphology suggested that the particle agglomerated, and the clusters of particles are all granular with the same average size. Agglomeration occur due to its natural state that is unstable. Subsequently, the formation of silica nanoparticles and their nanoscale size was confirmed. According to Daulay *et al.,* [19], it is possible to prevent agglomeration by altering the functional group on the surface of silica nanoparticles. Moreover, FESEM analysis was also conducted to obtain the grain size of the silica sand. The grain size and average grain size was measured by using bar scale as standard and Image-J software to measure the diameter of the particles. Thus, the average particle size is 337.56 nm.

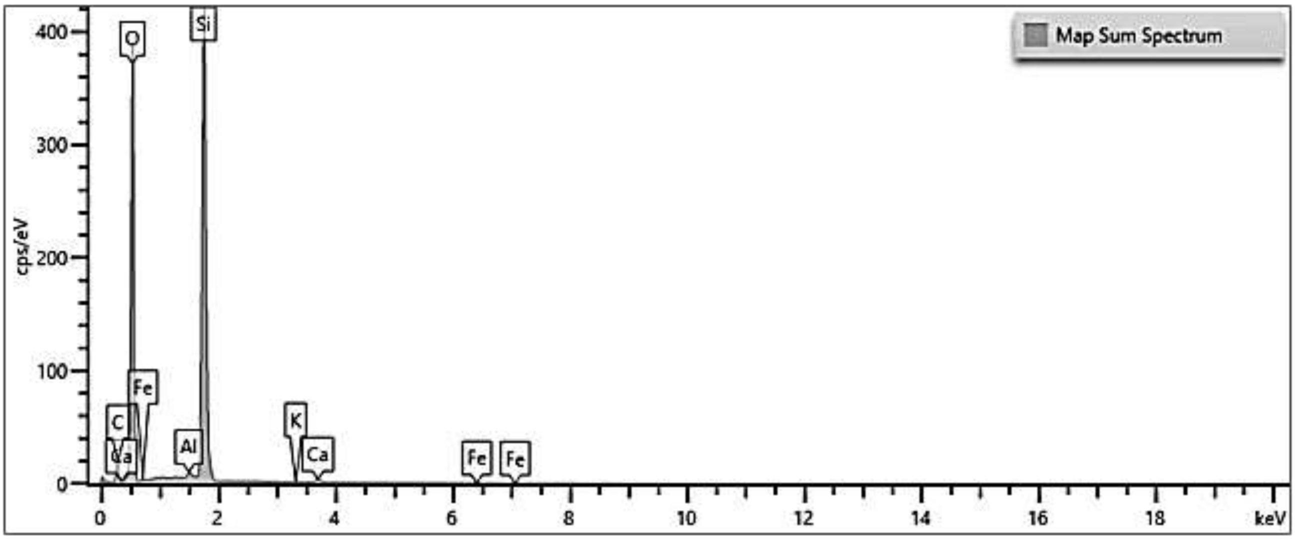


(a) (b)

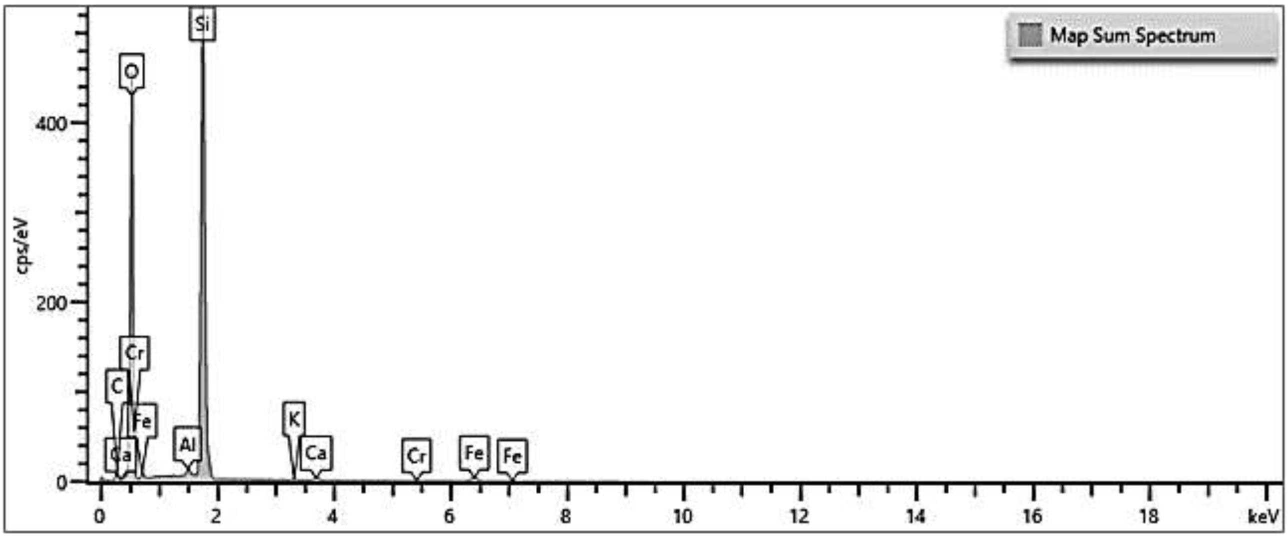
Fig. 8. FESEM image (a) Raw silica (b) Sintered silica

3.4 Elemental Analysis

Figure 9 shows the elemental analysis of raw silica sand and sintered silica sand. The element presence in both samples is Si, O, C, Fe, Al, Ca, Cr, K. According to research reported by Rampe *et al.,* [16], silica sand has a characteristic of having clear colour, consist of oxides and it is formed from weathered rock and contains silica compound. The example of the elements presence in the sample are SiO_2, Al_2O_3, CaO, Fe_2O_3 and K_2O as also reported by Rampe *et al.,* [16]. Since the main compound in the quartz sand is silica (SiO_2), thus the impurity materials presence in the quartz sand acts as a colouring agent that shows the percentage degree of purity of quartz sand can be estimated. Beach sand's purity varies according on its formation process as being reported by Silverstein *et al.,* [20]. According to Trisunaryanti *et al.,* [21], the high content of Silica indicates its potential to be used as a silica source.



(a)



(b)

Fig. 9. EDX pattern (a) Raw silica (b) Sintered silica

3.5 Microwave Characterization Analysis

The EM wave absorption performance can be determined by referring to the values of reflection loss (RL). Figure 10 shows the reflection loss of single and multilayer 3D printed polymer composite of frequency fixed at 4 to 8 GHz (C-band). The prepared 3D printed sample with only 5wt% loading amount and thickness fixed at 1 mm (single layer) and 2 mm (multilayer) were directly measured by using Vector Network Analyzer of one-port measurement and backed by metal plate. According to

Lee [22], the EMW reflectivity reduction can be correlated with the percentage of absorbed energy as shown in Table 2. The results obtained shows > 90% of the EM wave signal being absorbed (shown by the dotted line) by the materials at different resonance peak and at different frequency. For the single layer sample, 3D printed silica shows the best performance in the frequency range of 5.5 to 6.5 GHz with the minimum reflection loss reached -26 dB. However, there are existence of additional secondary and tertiary resonance peak at 6.94 GHz (RL = -6.51 dB) and at 7.81 GHz (RL = -1.79 dB) respectively. As for single layer 3D printed mill scale, the existence of the resonance peaks is at 4.52 GHz (RL= -2.73 dB), 6.19 GHz (RL= - 19.4 dB) and 7.99 GHz (RL = -4.32 dB).

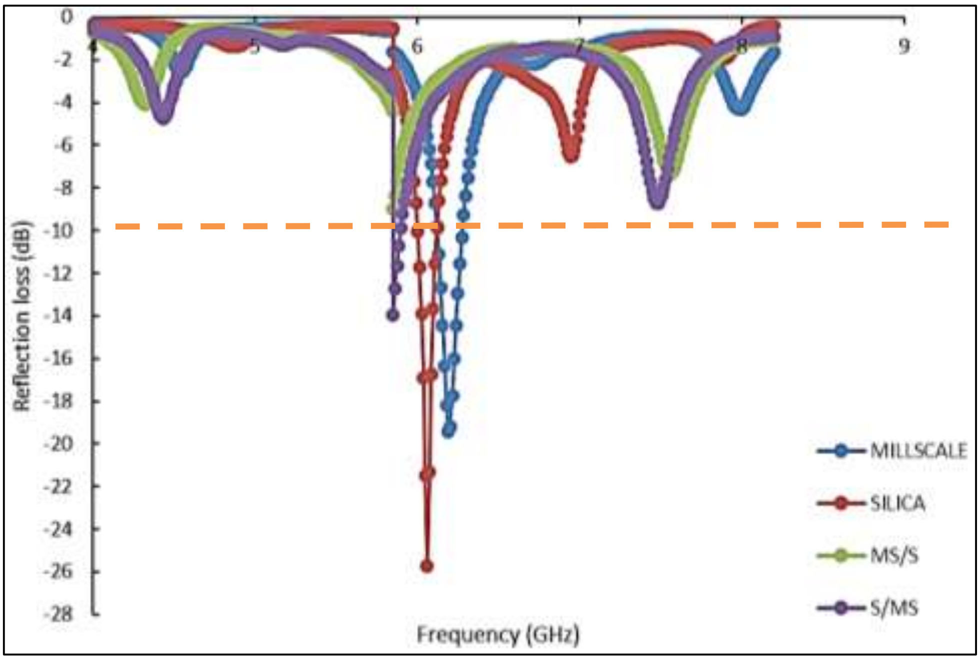


Fig. 10. Reflection loss performance of single and multilayer sample at frequency of 4-8 GHz (C-band)

Table 2
Relationship between reflectivity reduction and the absorbed energy [22]

Reflectivity reduction, dB	Absorbed energy, %
0	0
-3	50
-10	90
-15	96.9
-20	99
-30	99.9
-40	99.99

According to data recorded by Marpaung *et al.,* [12], silica have the ability to store the microwave energy since the imaginary part of the complex permittivity of silica are larger and it indicate stronger electric loss. Marpaung *et al.,* [12] also reported on the reflection loss of single layer silica SiO_2 with a thickness of 1 mm, 2 mm, 3 mm, 4 mm and 5 mm at frequency range of 8.2-12.4 GHz. The minimum RL values (less than -10 dB) of silica SiO_2 are -40 dB at 9.2 GHz with thickness of 4 mm. However, their research work does not state on the weight percentage of the filler loading amount. The reported results also show that double layer absorbing material showed different RL values than single layer of barium hexaferrite $BaFe_{12}O_{19}$ and silica SiO_2.

On the other hand, Yang *et al.*, [23] reported on preparing core-shell heterostructural nanocomposites with ferromagnetic nanowires as the core and silica as the shell. All chemicals were commercially purchased with additional liquid needed to prepare the solutions. They reported for sample filled with 50wt% with thickness of 7.25 mm; the reflection loss reaches -54.88 dB at 13.52 GHz. On the other hand, for frequency from 4 to 8 GHz, none of the thickness absorp the EMW with RL less than -10dB. Nevertheless, Tong *et al.,* [24] reported on carbonyl iron particles with 20% content, with RL between -15 dB to -25 dB of thickness 3 mm, 4 mm and 5 mm at frequency range of 4 to 8 GHz. Thus, it shows that the previous study obtained higher absorption performance, however with thicker and higher filler content. Higher filler content led to heavier materials that is not suitable for certain application. The limitation of having higher density and filling ratio limit their commercial applications, owing to excessive weight from metals (i.e. Co, Fe, Zn, etc) in the absorbing materials.

In this research work, 3D printed polymer composite sample of MS/S is referring to mill scale (MS) as the matching layer and silica (S) as the absorbing layer. On the other hand, as for S/MS is referring to silica (S) as the matching layer and mill scale (MS) as the absorbing layer. For both multilayer 3D printed polymer composite sample, it can be observed that there are three resonance peaks presence at different frequency with different RL values. As for MS/S, the resonance peaks presence at frequency of 4.31 GHz (RL = -4.01 dB), 5.91 GHz (RL = -9.23 dB) and 7.53 GHz (RL = -7.57 dB). On the other hand, the resonance peak obtained for S/MS are 4.43 GHz (RL = -4.75 dB), 5.85 GHz (RL = -14 dB) and 7.47 GHz (RL = -8.71 dB). By comparing all the resonance peaks obtained for both multilayer samples, it can be concluded that the resonance peak of both sample are slightly shift between each other showing that the resonance peak are almost at the similar frequency range. However, the reflection loss of S/MS of all the resonance peak showing that silica is the most suitable to be used as matching layer while, mill scale as an absorbing layer. This result is differed with result obtained by Marpaung *et al.,* [12] since the author research work is on double layer absorber composed of the absorption layer (Silica) and the matching layer (Barium hexaferrite).

Yusmaniar *et al.,* [25] reported on the performance of polymer composite at frequency of 8-12 GHz with the number of fillers to polymer is 20% to 80%. Based on the research work, they reported that the absorption of 20% magnetite composite at frequency ~10.6 GHz is -16.5 dB. On the other hand, with the amount of 20% silica as filler, the reflection loss reached -13 dB (~10.7 GHz). In addition, for mixture of 10% magnetite and 10% silica, the reflection loss reached -12.5 dB (~10.7 GHz). However, their research work is by using co-precipitation and solution casting method that is limited according to certain application and by using higher filler content. Several authors [26,27] reported that distinct absorptive strengths are observed in the data based on composite compositions.

Despite the widespread use of various EMW absorbing materials, the materials exhibit some limitations such as heavy weight, higher filler content, higher thickness and not suitable for certain applications. In contrast, this research work prepared lightweight, thin thickness, cost effectiveness by using industrial waste and silica from beach sand as the fillers. Moreover, the integration of additive manufacturing through the introduction of 3D-printed composite samples allows for customization in shape and size, while still maintaining excellent reflection loss (RL). This makes them suitable for the rapid advancement of portable electronic devices and the expanding applications of EMW absorbers. Additionally, these composites are cost-effective, widely available and involve a relatively simple manufacturing process. According to Latiff *et al.,* [28] by utilizing 3D printing technology, it will speed up production while cutting costs and it gives more impact to consumers demand. As it will saves both energy and time, the development of this 3D printing materials will be incredibly inventive and adaptable.

4. Conclusions

In conclusion, 3D printed EM wave absorber is successfully synthesized by introducing silica-based beach sand and industrial mill scale waste as filler. The synthesized 3D polymer composite sample results in better Electromagnetic Wave (EMW) absorption performance due to it being lightweight and having effective absorption for single and multilayer composite absorbers. Moreover, the single and multilayer 3D printed sample fabricated of different design, size and shape that meet the requirement for any application results in >90% in absorbing the EM wave interference. The investigation will be crucial in developing a new generation of single and multilayer 3D printed EM absorbing material based sustainable natural resource and industrial waste that gives best EMW performance of filler material to protect humans against harmful effects of electromagnetic radiation abundant in various industries. Therefore, this research work demonstrated that the prepared 3D printed polymer composites may have potential to be utilized at certain frequency range according to the electromagnetic absorbing applications.

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