JOURNAL OF CHEMICAL ENGINEERING AND INDUSTRIAL BIOTECHNOLOGY
E-ISSN: 0126-8139
VOLUME 9, ISSUE 2, SEPTEMBER 2023, 33 – 40
DOI: https://doi.org/10.15282/jceib.v9i2.9253

RESEARCH ARTICLE

Ferromagnetic Enhancement of Microcrystalline Cellulose via Chemical Reduction Method

S.H. Omar[1], R.M. Yunus[1]*, M.M.R. Khan[2], M.M. Saari[3]

[1] Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, Lebuhraya Persiaran Tun Khalil Yaakob 26300 Kuantan, Pahang, Malaysia.
[2] Petroleum and Chemical Engineering Programme Area, Faculty of Engineering, Universiti Teknologi Brunei, Gadong BE1410, Brunei.
[3] Faculty of Electric & Electronics Engineering Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, 26600 Pekan, Pahang, Malaysia.

ABSTRACT - Iron oxide nanoparticles (NPs) have potential in biological, biomedical, and environmental applications because of their characteristics such as magnetic susceptibility, stability and biocompatibility. However, it also has limitation, such as aggregation of magnetic NP. As a result, coating materials should be used to modify the particles' outer surface. In this paper, we focused on the synthesis of iron oxide by chemical reduction method and coating it with Fe(III) nitrate, polyvinylpyrrolidone (PVP) and hydrazine. In order to determine effective and economical usage conditions, the coating solution at two different concentrations were prepared. The effect of coating iron oxide with microcrystalline cellulose (MCC) was prepared at different concentrations of iron (III) nitrate on the nanomaterials with respect to morphological, thermal, magnetic susceptibility. A good morphology images of FeNp-MCC were proved by Scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Energy dispersive X-ray (EDX) spectra reveals the presence of carbon, oxygen and iron in the synthesized microparticles. TGA analysis showed iron material was successfully formed into the surface of MCC. Lastly, the magnetism results proved that cellulose is strongly interacting with magnetite nanoparticles.

ARTICLE HISTORY
Received : 23rd Jun. 2023
Revised : 4th Aug. 2023
Accepted : 7th Aug. 2023
Published : 30th Dec. 2023

KEYWORDS
Iron oxide nanoparticles
Chemical Reduction
Fe(III) nitrate
Microcrystalline cellulose
Magnetism

1.0 INTRODUCTION

Production of cellulose by green plants has received more attention recently owing to its renewable nature, such as wood, sugarcane bagasse and corn stover [1]. Cellulose materials such as nanocrystalline cellulose (NCC), microcrystalline cellulose (MCC) and nano-fibrillated cellulose (NFC) are widely used in environmental protection, water treatment, electronic components, biomedical, and other fields due to their biodegradability, biocompatibility, cost reduction and low toxicity [2]. Many materials were found to enhance cellulose material's characteristics and broaden its potential uses. Therefore, the addition of cellulose as a reinforcing agent is acknowledged as a current method that can result in a major change in the properties of cellulosic materials due to their strength and stiffness [3]. Cellulose are particularly useful as reinforcing agents in polymer composites where it can improve the mechanical properties of the material. However, a lot of researchers pointed out that their dispersion and alignment is a major weakness. Due to these reasons, cellulose fibre-reinforced composites have been less attractive for industrial production. Since better dispersion and alignment offer a unique approach to produce oriented structure and functional material with improved properties. A way to overcome this problem is a further surface modification on the cellulose before blending it with composite material. The development of cellulosic materials with benefits including simple preparation, low cost, high stability and easy separation from solution by an external magnetic field (H) is currently the focus of recent research [4].

According to T. Kimura, the magnetic field is a useful technique for processing weak magnetic materials [5]. When a feeble magnetic particle is subjected to a magnetic field, the particle receives a magnetic force so that it is pushed toward the location where the field strength is weak or strong, depending on its magnetic nature [1]. If a particle has a magnetic moment, it undergoes magnetic alignment. However, cellulose is not magnetically responsive (magnetism). There are several types of magnetic characters, which are paramagnetic, ferromagnetic, anti-ferromagnetic, and diamagnetic. Figure 1 illustrates the differences in the magnetic dipole moments with and without an external magnetic field (H) [6]. Paramagnetic atoms and molecules have a magnetic moment but are weakly attracted and affected by a magnet. However, ferromagnetic material is strongly attracted by magnets since it has the highest magnetic susceptibility and can be permanently magnetized, for example, iron (Fe), nickel (Ni), and cobalt (Co). A magnet strongly repels the dipoles in anti-ferromagnetic materials, reducing magnetic susceptibility. Diamagnetic material is weakly repelled by the magnetic field, which means it does not have a magnetic moment. This study used microcrystalline cellulose (MCC) due to its easy

***CORRESPONDING AUTHOR | R.M Yunus** | ✉ rmy@ump.edu.my

obtain commercially, low cost, non-toxicity and reusable. Thus, the best way to apply the diamagnetic microcrystalline cellulose (MCC) to a magnetic character is to combine it with a ferromagnetic material, such as iron particles [6].

Iron oxide nanoparticles (NPs) is considered the most attractive ferromagnetic material due to high saturation magnetization, biocompatibility, low toxicity, easy synthesis, and suitable particle shape and size [7]. Many researchers have utilized various synthesis methods to produce iron oxide nanoparticles in two different ways, physical and chemical methods, such as co-precipitation, sol-gel, micro emulsion, and hydrothermal technique [8]. In this research, the chemical reduction method is chosen to modify iron oxide nanoparticles using Fe(III) nitrates ($Fe(NO_3)_3.9H_2O$) as a starting material. In contrast, hydrazine and polyvinylpyrrolidone (PVP) were used as reducing agents and surfactants. According to the literature cited by K. Chou, the chemical reduction method was chosen because of its simplicity and low cost in synthesizing metal microparticles [9]. In this study, MCC was covered with iron nano particles at different concentrations to study the morphology, thermal and magnetic properties. SEM-EDX, TEM, TGA, FTIR spectra and AC magnetic susceptibility measurements of the FeNp-MCC were analyzed.

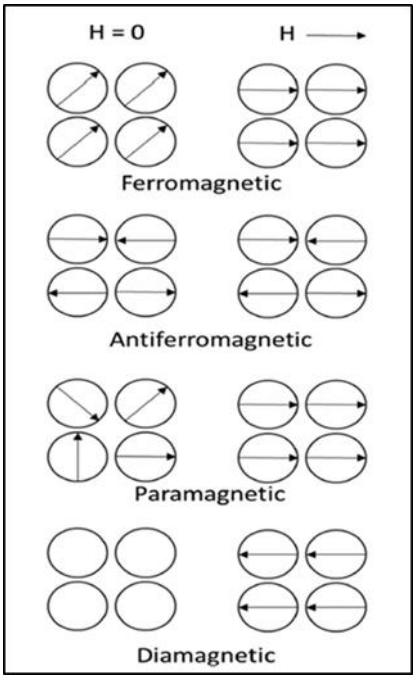

Figure 1. Illustration of the differences in the magnetic dipole moments with and without an external magnetic field (H)[6]

2.0 METHODS AND MATERIAL

2.1 Acid Hydrolysis Process

Microcrystalline cellulose (MCC) was purchased from Gardner global enterprise at Kuantan Pahang. Firstly, MCC was put in a two-necked flask connected by a condenser. The flask was equipped with a reflux system to prevent water loss by evaporation. Then, 64 % of sulphuric acid (H_2SO_4) was added into the solution at 45 ºC for 60 min. After that, deionized water was added at 5 ºC to stop hydrolysis. The mixture was then separated by centrifuging to abolish the acid solution and washed with distilled water until the water became clear. Finally, the sample was dried at 65 ºC using an oven until fully dried, and the sample was grinded by using a mortar and pestle for further processing.

2.2 Microcrystalline Cellulose (MCC) Coated with Iron Oxide Nanoparticles (NPs)

Effect of iron-coated cellulose, especially on the morphology of the iron nanoparticles, was observed by verifying the concentration of iron (III) nitrates which is 2.5 % and 5.0 % concentration. In order to reduce cost and toxicity, the appropriate iron concentration is important for the formulation of low concentration iron cellulose with controlled phase, morphology and magnetic properties. The existing method reported by Dadosh was modified slightly, and the experimental technique was used in this study [10]. In detail, dried cellulose was mixed with polyvinylpyrrolidone (PVP). Then, the sample with 200 ml of deionized water was sonicated for 20 min at room temperature. Next, the mixture was put in a two-necked flask, and different concentrations (2.5 % and 5.0 %) of iron (III) nitrate were added at 70 ºC for an hour. Following an hour of stirring, the temperature increased up to 100 ºC. Hydrazine was then gradually added to the solution in the flask until the solution's colour changed. Then, the solution was stirred continuously at room temperature overnight to cool down the solution. The sample was then filtered and repeatedly washed with methanol and deionized water. Lastly, the sample was dried at 80 ºC for 24 hours.

2.3 Characterization Test

Many different techniques can be used to characterize the iron coated cellulose, such as scanning electron microscopy with energy dispersive X-ray (SEM-EDX), transmission electron microscopy (TEM), thermal analysis (TGA), infrared spectroscopy (FTIR) and alternating current (AC) magnetic susceptibility

2.3.1 Morphology Analysis

The morphology of the composite surface was examined by scanning electron microscopy with energy dispersive x-ray (SEM-EDX) mapping and transmission electron microscopy (TEM). SEM-EDX analysis was conducted using a (Hitachi TM 3030 Plus, Japan) at 5 kV high vacuum conditions. All specimens were sputter coated with gold before the examination. An energy-dispersive X-ray analyzer (EDX) was also used to identify iron, carbon, and oxygen elements in magnetite microparticles. Next, transmission electron microscopy (TEM), JEOL JEM 1400, was used to measure the morphology of the iron-coated cellulose (FeNp-MCC). The sample of FeNp-MCC was measured in colloidal form with distilled water as a solvent.

2.3.2 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was performed to analyze the thermal properties of iron microcrystalline cellulose using a TGA analyzer (Hitachi/STA7000, Japan). The TGA thermograms was obtained by heating the sample under nitrogen flow from room temperature to 700 ºC at 10 ºC/min (heating rate).

2.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The vibrational spectroscopic approach was utilised to determine the specific functional groups within catalyst sample using FTIR analysis. FTIR, Perkin-Elmer USA spectra for MCC and FeNp-MCC were obtained with a 400 – 4000 cm^{-1} wavenumber range.

2.3.4 Alternating Current (AC) Magnetic Susceptibility

Quantum Design MPMS-S5 Squid magnetometer with an AC susceptibility option was used to characterize magnetic susceptibility [11]. The measurements were performed with an AC amplitude of 0.05 mTpp, at the temperature of 26 ºC and a frequency of 5 Hz – 100 kHz. The samples were prepared in water and epoxy suspension to check and stop Brownian relaxation.

3.0 RESULTS AND DISCUSSION

The morphology of samples via SEM-EDX mapping and TEM were examined to understand changes in the iron microparticle performance.

3.1 Scanning Electron Microscopy with Energy Dispersive X-ray (SEM-EDX)

Scanning electron microscopy with energy dispersive X-ray analysis was used to examine the samples' surface morphology and element composition (SEM-EDX). SEM images of modified microcrystalline cellulose (MCC) by iron oxide nanoparticles (NPs) are shown in Figure 2. SEM images showed that the nanocomposite's surface changes when the iron concentration increases. The sample for the MCC was found to have a fibril-like structure. For the hybrids, iron-coated cellulose shows the matrix fibril character and the presence of crystal deposition at their surface, as indicated by the circle that represents the deposition of iron cellulose. From the findings that support Vallejo's research, it was also noted that the MCC sample had a fibril-like shape [12].These results indicate that nanoparticles obtained by 5 % iron cellulose produced more iron cellulose deposition than 2.5 % iron cellulose.

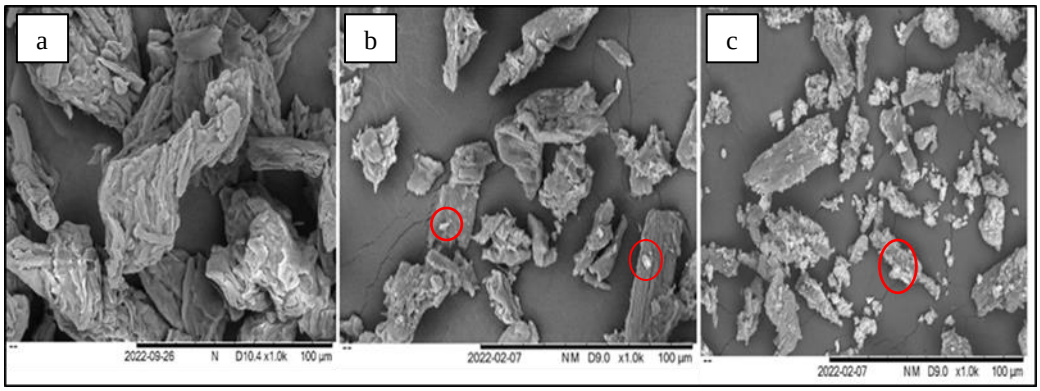

Figure 2. SEM images for (a) MCC at 100 $\boldsymbol{\mu m}$(b) 2.5 % iron-coated cellulose at 100 $\boldsymbol{\mu m}$ (c) 5.0 % iron-coated cellulose at 100 $\boldsymbol{\mu m}$

In addition to the SEM images, the energy dispersive X-ray (EDX) analysis spectrum of $Fe(NO_3)_3.9H_2O$ nanoparticles is shown in Figure 3 and figure 4 respectively. According to the EDX spectrum, the cellulose structures coated with iron

consist of ferum (Fe), oxygen (O_2), and carbon (C). However, it was necessary to coat the sample with a thin layer of gold (Au) to prevent surface charging and create a homogeneous surface for imaging and analysis [13]. Therefore, a small amount of Au is present originated from the SEM grid of the sample holder. From the analysis of the results, the main constituents of the composition of the prepared material produced are carbon, followed by oxygen and iron elements from the synthesis of iron oxide. However, 5.0 % iron coated cellulose produced more ferum than 2.5 % iron-coated cellulose. The results of SEM-EDX, show that the sample is composed of a nanocomposite of cellulose and iron oxide indicating successful synthesis of iron-coated cellulose.

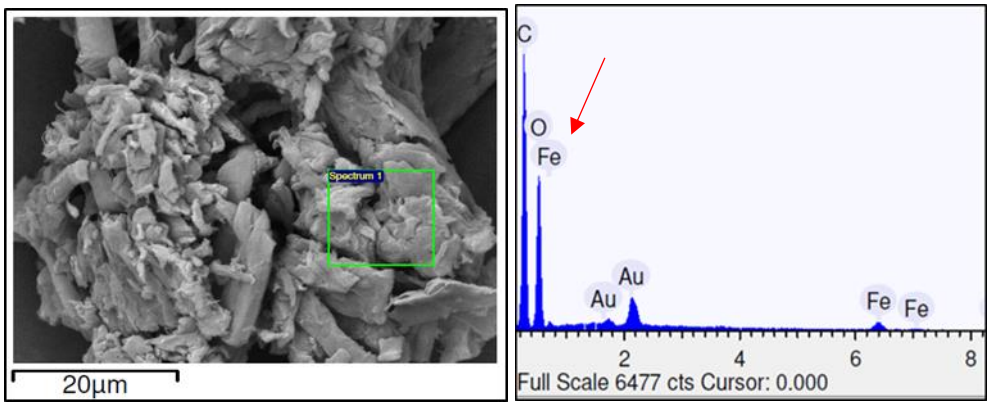

Figure 3. SEM-EDX images for 2.5 % iron-coated cellulose at 20 $\boldsymbol{\mu m}$

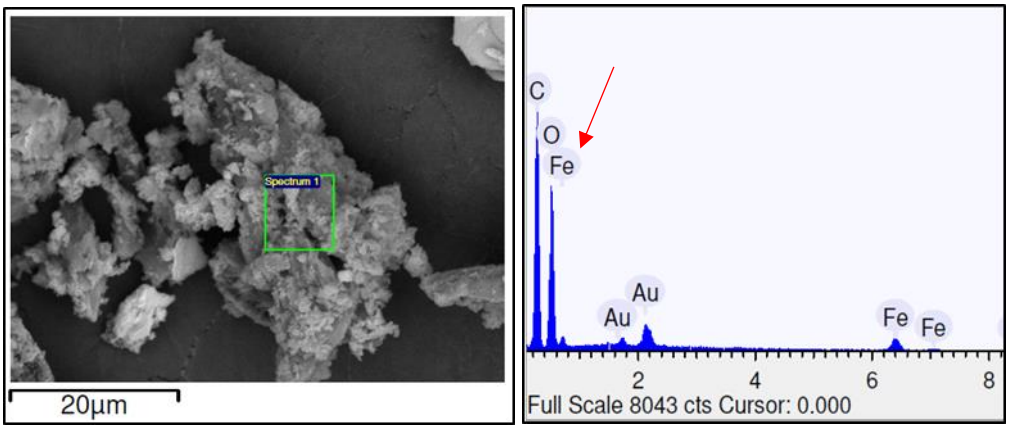

Figure 4. SEM- EDX images for 2.5 % iron-coated cellulose at 20 $\boldsymbol{\mu m}$

3.2 Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) was used to examine the morphology of cellulose materials, the quality of their surface coating and their dispersion. Figure 5 displays images of iron microparticles coated with different amounts of iron (III) nitrates (2.5 % and 5.0 %). TEM images showed agglomerates of small grains and some dispersed microparticles. The images of the samples show well-dispersed, more or less spherical particles. The figure showed that iron oxide nanoparticles (NPs) was uniformly covered on the surface of microcrystalline cellulose (MCC), and the obtained iron-coated cellulose exhibited good morphology. It was found that iron oxide is well distributed in iron cellulose (2.5 % and 5.0%). However, some agglomerates are observed in the microstructures when the iron oxide concentration reaches 5.0 % compared to 2.5 % iron-coated cellulose. This result agrees with similar results on the effects of morphology on magnetite cellulose nanofibrils [14].

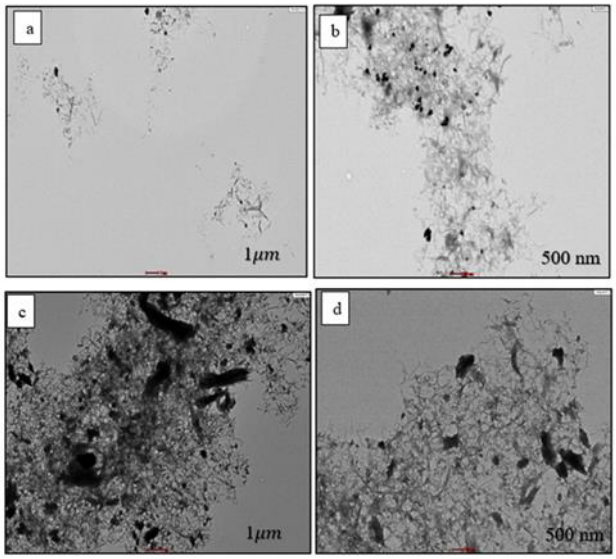

Figure 5. TEM images for (a) 2.5 % iron-coated cellulose at 1 $\boldsymbol{\mu m}$ (b) 2.5 % iron-coated cellulose at 500 nm (c) 5.0 % iron-coated cellulose at 1 $\boldsymbol{\mu m}$ (d) 5.0 % iron-coated cellulose at 500 nm

3.3 Thermogravimetric analysis (TGA)

"Thermogravimetric analysis (TGA) is a method for thermal analysis that measures how the physical and chemical properties of materials change as a function of decreasing temperature (with constant heating rate) or as a function of time" [15]. The thermal analysis curve of microcrystalline cellulose (MCC) and iron-coated cellulose at different iron concentrations is represented in Figure 6. The initial degradation temperature (T_{on}) and maximum weight loss (T_{max}) at different temperatures have been analyzed. Figure 6 shows that 5.0 % and 2.5 % iron cellulose powder have better thermal stability than MCC powder. The weight loss temperature decreased slightly due to incorporating ferromagnetic materials in the MCC. From the result, the maximum weight loss in 5 and 2.5 % iron coated cellulose was observed at around 300 º C. M-G.Ma et al. reported that the weight loss in the region 200 -500 ºC can be assigned to the thermal degradation and complete decomposition of cellulose in the composite [16]. At the same time, pure MCC showed a temperature of 360 º C. The results are comparable to P. studies's that reported the common thermal breakdown of cellulose occurs between 250 ºC to 375 ºC [17]. TGA study confirmed the difference in thermal behavior between pure MCC and modified MCC.

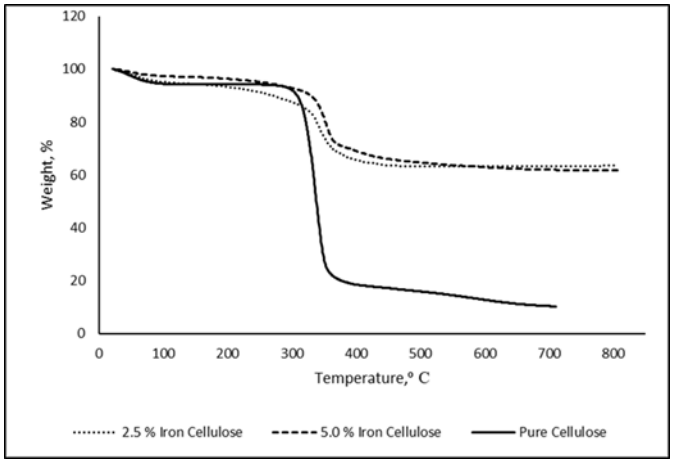

Figure 6. TGA curve of iron-coated cellulose at different concentration

3.4 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) of the iron-coated cellulose at different concentrations (2.5 % and 5.0 %) is depicted in Figure 7. The full spectra were obtained from the 400 to 4000 cm^{-1} range. 5.0 % iron-coated cellulose cellulose exhibits well-defined peaks at 559,879, 1751, and 3001 cm-1. The observed FTIR results for 2.5 % iron cellulose showed various peaks at 567,891,1061 and 1624 cm-1. The appearance of distinct peaks (559 and 567 cm-1) is due to the presence of iron-oxygen (Fe-O), which confirmed the existence of ferric ions and the union with oxygen ions of a hydroxyl group of cellulose to produce iron oxide [18]. In agreement with literature by M.Yadav et al., these results indicated a strong interaction between Fe_3O_4 and cellulose [18]. In addition, the small peaks at 879 and 891 cm-1 are due to the presence of the nitrate group. Moreover, the band at 1751 and 1624 are due to the stretching vibration of the alcoholic hydroxyl (C-O). These spectra also present the vibrational mode characteristics of the organic structure of cellulose. Other

than that, the intensity of the 3001 cm^{-1} peak, which is related to the O-H groups of cellulose, was justified by the non-availability of the hydroxyl group and their interaction with iron ions [19]. The FTIR data revealed that the synthesized micro-particles are iron oxide without major impurities. Lastly, these data demonstrated that the surface of magnetic microparticles had been covered with MCC.

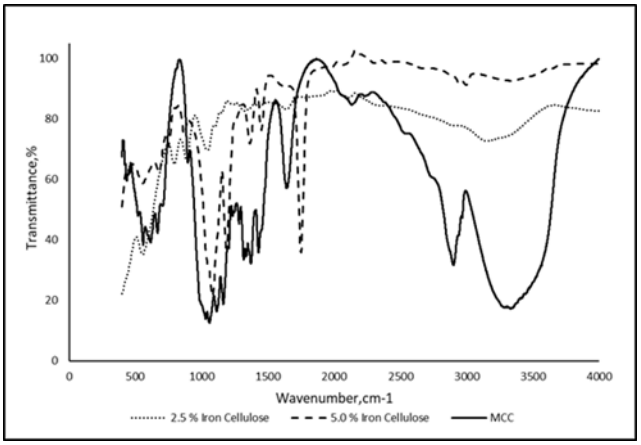

Figure 7. FTIR spectra of iron-coated cellulose at different concentrations

3.5 Alternating Current (AC) Magnetic Susceptibility

The produced dark brown reddish iron oxide micro-particles are paramagnetic in nature and strongly attracted to the magnetic bar. Figure 8 represents the frequency-dependent real and imaginary susceptibility of the synthesized FeNp-MCC in the water and epoxy suspension. This approach applied an alternating current magnetic field (the excitation) to the sample and recorded the subsequent alternating current magnetization (the response). Figure 8 showed a negligible difference between water and epoxy suspension; no significant Brownian relaxation was observed. It is because the iron oxide particle is fixed in the cellulose matrix, producing a larger effective particle size bigger than 500 nm. A similar pattern is found by F. ludwig and H.Remmer et al., the size of the iron oxide core can be estimated greater than 500 nm, in which the Neel relaxation time t_N will exponentially increase in this region [20].The trend starts to decay below 10 Hz. The current instrument's measurement below 5 Hz is extremely difficult due to the high noise level in the lower region. Besides that, the 5 % sample has almost the same characteristics as the 2.5 % sample. However, the 5 % sample has a higher volume number of smaller particles than the 2.5 % sample. The signal from the sample indicates good ferromagnetic properties, but the Neel relaxation of larger particles dominates the relaxation.

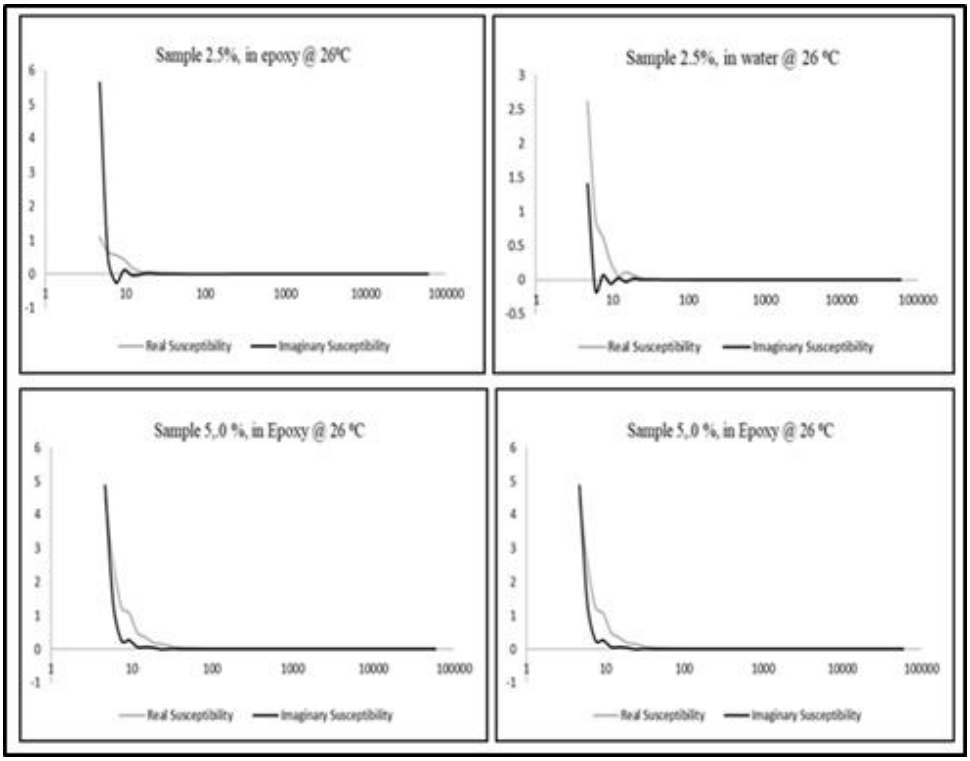

Figure 8. AC magnetic susceptibility of iron coated cellulose in water and epoxy suspension

4.0 CONCLUSION AND RECOMMENDATION

In this study, iron-coated cellulose was successfully synthesized by the chemical reduction method. Two different conditions are used in this study to produce iron-coated cellulose: iron (III) nitrate concentrations of 2.5 % and 5.0 %. Generally, adding iron oxide to cellulose enhances the nanomaterial's morphology, thermal stabilities, and magnetic properties. From SEM and TEM images, it was confirmed that the good mixing of iron oxide in cellulose matrix and Fe_3O_4 is well distributed in cellulose. TGA result indicates the weight loss temperature for modified cellulose was decreased slightly due to the incorporation of ferromagnetic material. In addition, FTIR indicated that the surface of magnetite particles was covered with MCC. The magnetic susceptibility response from the modified sample showed good ferromagnetic properties, but the Neel relaxation of larger particles dominated the relaxation. To overcome this problem, further work will focus on preparing modified cellulose using nano-crystalline cellulose (NCC) as the main ingredient to get smaller particles and better relaxation in AC magnetic susceptibility. It can be concluded, 5.0 % iron (III) nitrate concentration gives the best iron coated cellulose than 2.5 % concentration.

5.0 CONFLICT OF INTEREST

The authors declare no conflicts of interest.

6.0 AUTHORS CONTRIBUTION

S.H. Omar (Conceptualization; Visualisation; Writing-original draft)

R.M. Yunus (Supervision; Review)

M.M.R. Khan (Supervision; Review)

M.M. Saari (Software)

7.0 ACKNOWLEDGEMENTS

This work was supported by the Malaysia Ministry of Higher Education under the Fundamental Research Grant Scheme (FRGS) No. FRGS/1/2018/TK05/UMP/01/2 (University reference RDU190138).

8.0 REFERENCES

[1] F. Kimura and T. Kimura, “Magnetic alignment and patterning of cellulose fibers,” *Sci. Technol. Adv. Mater.*, vol. 9, no. 2, pp. 1–4, 2008, doi: 10.1088/1468-6996/9/2/024212.

[2] Mehran Alavi, “Modifications of microcrystalline cellulose (MCC), nanofibrillated cellulose (NFC), and nanocrystalline cellulose (NCC) for antimicrobial and wound healing applications,” *e-Polymers*, vol. 19, no. 1, pp. 103–119, 2019.

[3] Y. Zhao, J. Qiu, H. Feng, M. Zhang, L. Lei, and X. Wu, “Improvement of tensile and thermal properties of poly(lactic acid) composites with admicellar-treated rice straw fiber,” *Chem. Eng. J.*, vol. 173, no. 2, pp. 659–666, 2011, doi: 10.1016/j.cej.2011.07.076.

[4] L. Chen, S. Sharma, R. E. Darienzo, and R. Tannenbaum, “Decoration of cellulose nanocrystals with iron oxide nanoparticles,” *Mater. Res. Express*, vol. 7, no. 5, p. 55003, 2020, doi: 10.1088/2053-1591/ab8a82.

[5] T. Kimura, “Study of the effect of magnetic fields on polymeric materials and its application,” *Polym. J.*, vol. 35, no. 11, pp. 823–843, 2003, doi: 10.1295/polymj.35.823.

[6] T. Nypelö, “Magnetic cellulose: Does extending cellulose versatility with magnetic functionality facilitate its use in devices?,” *J. Mater. Chem. C*, vol. 10, no. 3, pp. 805–818, 2022, doi: 10.1039/d1tc02105b.

[7] S. Xu and T. R. Lee, “applied sciences Fe 3 O 4 Nanoparticles : Structures , Synthesis , Magnetic Properties , Surface Functionalization , and,” 2021.

[8] W. Wu, Q. He, and C. Jiang, “Magnetic iron oxide nanoparticles: Synthesis and surface functionalization strategies,” *Nanoscale Res. Lett.*, vol. 3, no. 11, pp. 397–415, 2008, doi: 10.1007/s11671-008-9174-9.

[9] K. Sen Chou and C. Y. Ren, “Synthesis of nanosized silver particles by chemical reduction method,” *Mater. Chem. Phys.*, vol. 64, no. 3, pp. 241–246, 2000, doi: 10.1016/S0254-0584(00)00223-6.

[10] T. Dadosh, “Synthesis of uniform silver nanoparticles with a controllable size,” *Mater. Lett.*, vol. 63, no. 26, pp. 2236–2238, 2009, doi: 10.1016/j.matlet.2009.07.042.

[11] M. M. Saari *et al.*, “A benchtop induction-based AC magnetometer for a fast characterization of magnetic nanoparticles,” *Eng. Res. Express*, vol. 4, no. 2, p. 25047, 2022, doi: 10.1088/2631-8695/ac78c8.

[12] M. Vallejo *et al.*, “Recovery and evaluation of cellulose from agroindustrial residues of corn, grape, pomegranate, strawberry-tree fruit and fava,” *Bioresour. Bioprocess.*, vol. 8, no. 1, 2021, doi: 10.1186/s40643-021-00377-3.

[13] S. A. Leslie and J. C. Mitchell, “Removing gold coating from sem samples,” *Palaeontology*, vol. 50, no. 6, pp. 1459–1461, 2007, doi: 10.1111/j.1475-4983.2007.00718.x.

[14] T. C. Breijaert *et al.*, “Self-assembly of ferria – nanocellulose composite fibres,” *Carbohydr. Polym.*, vol. 291, no. May, p. 119560, 2022, doi: 10.1016/j.carbpol.2022.119560.

[15] H. M. Ng, N. M. Saidi, F. S. Omar, K. Ramesh, S. Ramesh, and S. Bashir, "Thermogravimetric Analysis of Polymers," *Encycl. Polym. Sci. Technol.*, no. November, pp. 1–29, 2018, doi: 10.1002/0471440264.pst667.
[16] M. G. Ma, J. F. Zhu, S. M. Li, N. Jia, and R. C. Sun, "Nanocomposites of cellulose/iron oxide: Influence of synthesis conditions on their morphological behavior and thermal stability," *Mater. Sci. Eng. C*, vol. 32, no. 6, pp. 1511–1517, 2012, doi: 10.1016/j.msec.2012.04.033.
[17] P. Studies, "Synthesis and Characterization of Iron Oxides onto Cellulose Supports for Adsorption of Roxarsone," no. March, 2017.
[18] M. Yadav, "Study on thermal and mechanical properties of cellulose/iron oxide bionanocomposites film," *Compos. Commun.*, vol. 10, no. January, pp. 1–5, 2018, doi: 10.1016/j.coco.2018.04.010.
[19] I. M'barek *et al.*, "Nanocellulose synthesis from Tamarix aphylla and preparation of hybrid nanocellulose composites membranes with investigation of antioxidant and antibacterial effects," *Sep. Purif. Technol.*, vol. 292, no. February, 2022, doi: 10.1016/j.seppur.2022.120815.
[20] F. Ludwig and H. Remmer, "Rotational dynamics of magnetic nanoparticles in different matrix systems," *Magn. Hybrid-Materials Multi-scale Model. Synth. Appl.*, pp. 323–350, 2021, doi: 10.1515/9783110569636-013.