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Magnetic and Structural Analysis of Magnetorheological Foam Fabricated by Constraining Volumes of Foaming Process

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ABSTRACT

Magnetorheological (MR) foam has become a topic of research interest due to its potential breakthrough in smart soft robotic applications, due to its versatility, high flexibility and durability. In fact, it has controllable properties that could be actively changed, reversibly by manipulating external magnetic fields. The process of magnetizing MR foam is highly dependent on the incorporation and distribution of magnetic particles (CIPs) in the porous structure of the foam. In fact, the resultant properties would be affected by the structural arrangement of the material. Therefore, this study provides an insightful analysis on the microstructure of MR foams and its resultant magnetic properties, after undergoing different constrained volumes of foaming process. The magnetizations of fabricated samples were determined via vibrating sample magnetometer (VSM) and the morphological characteristic was carried out by using atomic-force microscopy (AFM). The results demonstrate that the magnetic properties of MR foam increased as constrained volumes were applied during foaming process, from free foaming to 50% constrained volume. This indicates the improvement in the magnetic response of MR foams, and higher magnetic saturation due to more localized CIPs in the structure of MR foams. AFM analysis then further supports these findings by showing distinct structural changes in the porous structure of MR foams as the constrained volume during foaming decreased. Consequently, a more compact structure with improved distribution of CIPs has been acquired, which enhances the connectivity between the CIPs and contributes to a more robust magnetic network when MR foam is exposed to applied magnetic fields. This research provides a foundation for further exploration of MR foams, as the improvements are critical for future applications, particularly in robotic fields, requiring high-performance MR foams.

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1. Introduction

Magnetorheological (MR) foam represents a cutting-edge development in the field of smart materials that offering a lightweight, flexible and tuneable properties under the influence of magnetic fields. MR foam shall render a desirable interest for various advanced applications such as semi-active soft robotic grippers, flexible sensors, adaptive actuators, and potentially to be apply for automotive applications such as car cabin insulation for noise reduction and chassis components for vibration damping and absorption. This is due to its ability to bridge the gap between fixed properties of current available materials and those that are adaptable and adjustable in real-time environments [1,2]. The foundation of MR foam lies in the interplay between the flexible polyurethane foam (PU) as a matrix and the embedded magnetic particles, typically carbonyl iron particles (CIPs) in the structure of the foam. The sensitivity of the magnetic properties of MR foam, including magnetic saturation, magnetic remanence and coercivity are primarily influenced by the concentration and distribution of CIPs within the matrix per unit volume [3]. In fact, magnetic properties plays a crucial role in ensuring strong magnetic response under the presence of magnetic field [4-6]. Generally, magnetic saturation refers to the maximum magnetization that the MR foam can achieve upon an external magnetic field, while magnetic remanence is the residual magnetization remaining after the external field is removed [7,8]. On the other hand, coercivity is the measure of the resistance of the magnetic material to changes in magnetization [7,8]. These properties are essential for determining the performance and potential applications of MR foams [9,10].

Up to date, CIPs have been commonly used as magnetic particles in MR foam [11-16]. While these particles exhibit high magnetic saturation, high permeability, and low remanence magnetization, the overall properties of MR foam are highly dependent on its internal structure. As a consequence, previous studies [12,15-19] have extensively explored the fabrication and characterization of MR foams, indeed focusing on enhancing its rheological and mechanical properties. Recent advancements have introduced innovations such as constraint volume foaming processes for the fabrication of MR foam. Through this fabrication method, the foaming process experienced a restricted expansion and constrained environments in tandem to enhance the uniformity and compact distribution of CIPs within the foam structure. As previously reported [14], this method has shown a promising enhancement in the storage modulus and overall structural performance of MR foam. It was reported that the introduction of constraint volume foaming process has prominently enhance the structural properties and a notable compact distribution of CIPs is expected to be the reason behind this enhancement. However, despite the significant contribution, observation on the effect of constraint foaming processes on changes in magnetic properties have not been fully explored.

Nevertheless, several researchers have studied on the magnetic properties of CIPs in various kind of solid MR materials [4,15,16,20,21]. Zainudin *et al.,* [4] has reported a study on the magnetic properties of MR elastomer (MRE) fabricated by isotropic and anisotropic curing condition. It was reported that the coercivity magnetic hysteresis showed a similar behaviour for both curing conditions explaining that the properties of CIPs as soft magnetic particles was not affected by its distribution in MRE samples. Instead, the difference in magnetic performance of MRE was mainly attributed to the particle alignments where anisotropic MRE displayed higher magnetic saturation compared to isotropic MRE. This phenomenon was in line with previous finding who reported that the magnetic saturation was affected by concentration of CIPs incorporated in MRE samples [22]. Higher concentration of CIPs displayed higher magnetic saturation due to a more ability of CIPs in forming chain-like structures under the presence of magnetic field. Indeed, the increase of magnetic particle concentrations significantly enhanced the overall magnetic properties due to more frequent

interfaces between particles [20]. On the other hand, similar phenomenon for MR foam was reported by Rizuan *et al.,* [16] where the study reported that similar pattern of hysteresis loop was observed for all MR foam samples regardless the concentration of CIPs particles. Admittedly, as discussed by Muhazeli *et al.,* [15] and Riesgo *et al.,* [21], the increased quantity of CIPs enhanced the magnetic properties of MR foam due to higher availability of magnetic moment in the materials. It is worthwhile to note that the magnetic properties were influenced by the magnetic interaction in the struts of MR foam samples. Higher magnetic saturation causes a stronger magnetic dipole attraction and as the results, an enhancement of rheological and mechanical properties were observed in the MR foam.

In this context, the present research aims to delve deeper into the magnetic properties of MR foams, examining how constraint volume foaming process affects the distribution of CIPs and influenced its magnetic properties. By understanding these relationships, researchers able to pave the way for innovations that enhance performance and expand the utility of these remarkable MR foam. This study not only contributes to the fundamental knowledge of MR foam but also holds the potential to revolutionize fields that require adaptive and responsive materials especially those who requires precise control of rheological, mechanical and magnetic properties.

2. Methodology

2.1 Materials and Sample Preparation

In general, the fabrication of MR foam requires two primary components: the matrix and the magnetic particles. The matrix material used for the MR foam samples was flexible polyurethane (PU) in liquid form, supplied by Smooth-On, Inc., USA. The PU foam was predominantly a blend of two parts, polyol and isocyanate. Specifically, polyether polyol (PPG)-based triol served as a chemical agent with a density of 1.03 g/ml, while 4,4'-methylene bis(phenyl isocyanate) acted as the chemical reactant, aiding in the formation of gas bubbles during the foaming process, with a density of 1.00 g/ml. To impart magnetic responsiveness, carbonyl iron powder (CIP) was utilized as the magnetic particles because it offers several advantages including high saturation magnetization, excellent permeability and low remanent magnetization, making them ideal for enhancing the magnetic responsiveness of MR foams [11,14-16]. This OM grade CIPs was characterized to have spherical shapes with an average diameter ranging from 3 to 5 µm and it was purchased from BASF, Ludwigshafen, Germany, and had a density of approximately 7.87 g/ml.

The experimental procedure for sample preparation involved the following steps. Figure 1 illustrates the procedure of the fabrication process, and the details of MR foam samples were listed down in Table 1. A free foaming MR foam was prepared with a fixed 75 wt.% of CIPs via an in-situ polymerization method under isotropic curing conditions to set as the benchmark for comparison purposes in this study. Firstly, the magnetic particles were added with polyol and stirred for 20 seconds using a mechanical stirrer at the speed of 550 rpm. Subsequently, isocyanate was added to the mixture and stirred at the same speed and duration. The final mixture was promptly poured into a 3-cm diameter cylindrical polyvinyl chloride mold for the foaming process to occur and then was left to cure at room temperature of 25°C for the duration of 24 hours. This free foaming MR foam is labelled as sample A. On a side note, the height of MR foam produced was observed and set as the maximum mould height for the 100% volume for the constraint volume foaming method. Indeed, it was conducted multiple times to ensure reliable height measurements were obtained. Differ from free foaming fabrication method, the constraint volume foaming method requires the mould to be immediately covered after the final MR foam mixture was poured into the mould. This was to create a constrained foaming environment for the MR foam sample. The sample was labelled as sample B

and then left at the same condition and duration as mentioned in the earlier steps. Lastly, the constraint volume foaming experiment was repeated with the mold length reduced by 25% and 50% from its original length. These samples were known as sample C and D respectively.

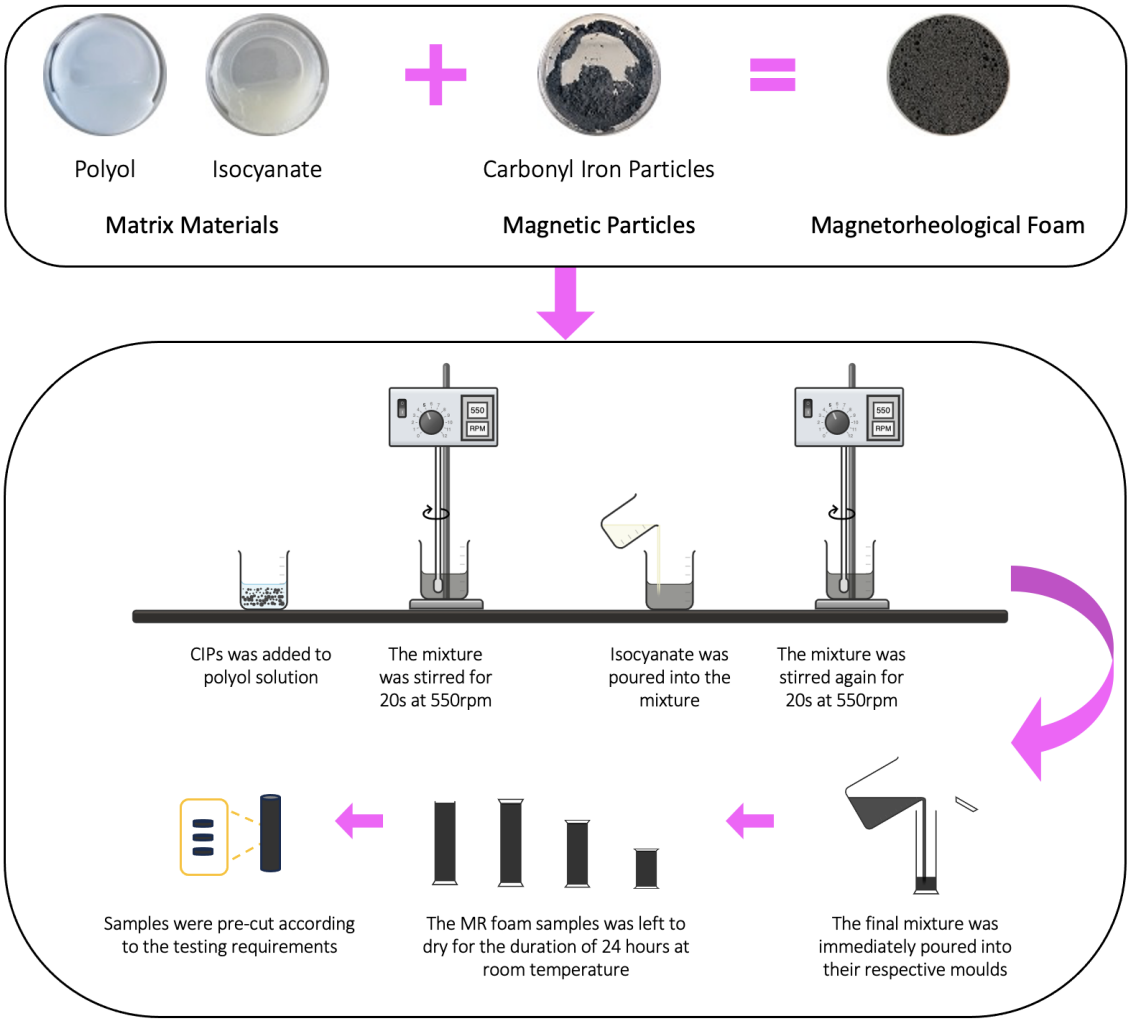


Fig. 1. Procedure of MR foam fabrication

Table 1
Details of the fabricated MR foam samples

Sample name	Remarks
A	Free foaming
B	100% volume of constrained foaming
C	75% volume of constrained foaming
D	50% volume of constrained foaming

2.2 Sample Analysis and Characterization

The magnetic properties of the fabricated MR foam samples were analyzed using a Vibrating Sample Magnetometer (VSM) from Lakeshore, USA, Model 7404 Series. This equipment is equipped with a computer running EasyVSM software (Version Easy VSM 20170412-01) and includes a Microsense VSM Vibrator Controller for data recording, a magnet coil, and a sample holder. Firstly, the MR foam samples were pre-cut into cubic shapes with dimensions of (4 × 4 × 4) mm, and the

weights were measured using an analytical weighing balance. The samples were then placed in a sample container and mounted on the tip of a rod sample holder that vibrated continuously during the analysis. The samples were wrapped in polytetrafluoroethylene (PTFE) to protect from displaced during testing and it was then positioned between two electromagnetic coils that generated magnetic fields ranging from -14 kA/m to 14 kA/m. Any changes in the magnetic fields were detected by the coil and expressed through the magnetization curve, also known as hysteresis curves of magnetization (M) versus magnetic field (H). Finally, the magnetic behavior of the fabricated MR foam samples, in terms of magnetic saturation (M_s), coercivity (H_c), and remanence (M_r), was obtained from this testing. On the other hand, a morphological analysis was conducted using an atomic force microscopy (AFM), Park FX40, an advance-automated AFM provided by Park System, Suwon, South Korea in order to study the microphase structure of the fabricated MR foam samples. This observation was crucial for evaluating the 3D surface features of MR foams prepared with different constraint volumes, within a predetermined scan region of 10 nm × 10 nm, at a scan rate of 0.14 Hz. The tapping mode analysis was conducted using an aluminum backside-coated cantilever, with a tip length of 10-15 nm and a radius of less than 10 nm. Consequently, Park Systems XEI software was used to create microphase images of a localized area of 20 µm × 20 µm obtained from the observation. All of the testing was conducted at room temperature of 25°C.

3. Results

3.1 Magnetic Properties Analysis

The incorporation of different constraint volumes of foaming process towards the fabrication of MR foams is believed to have significant influence to its magnetic properties which resulting to the enhancement of the properties of MR foam [14]. The magnetic properties of the fabricated MR foam samples were evaluated in terms of magnetization saturation (M_s), remanence magnetization (M_r) and coercivity (H_c), under the influence of magnetic field ranges from -14 kA/m to 14 kA/m at ambient temperature of 25°C. The magnetization curves of all MR foam samples are depicted in Figure 2 and the details regarding magnetic properties are listed in Table 2. Overall, the magnetization curves of all MR foam samples portray narrow hysteresis loops, which coincide to the soft magnetic characteristics of CIPs that has high magnetic saturation, low remnant and low coercive magnetic field [4,23-26]. Therefore, the remanence magnetization (M_r) for all MR foam samples is negligible. This finding is in line with previous studies who reported similar trend of magnetic curves for materials contained CIPs such as MR elastomer (MRE) and MR grease (MRG) [8,22,27-29]. Initially, the graph shows a constant value at low magnetic field intensity stipulate the CIPs has no response with no magnetic field applied. Then, the loop increased dramatically with increasing magnetic field intensity, up and until it reaches a certain intensity value, the slope levelled off again indicate that it has reached its saturation condition as the magnetic moment is assumed to be fully aligned according to the magnetic field direction [8,22,27-29].

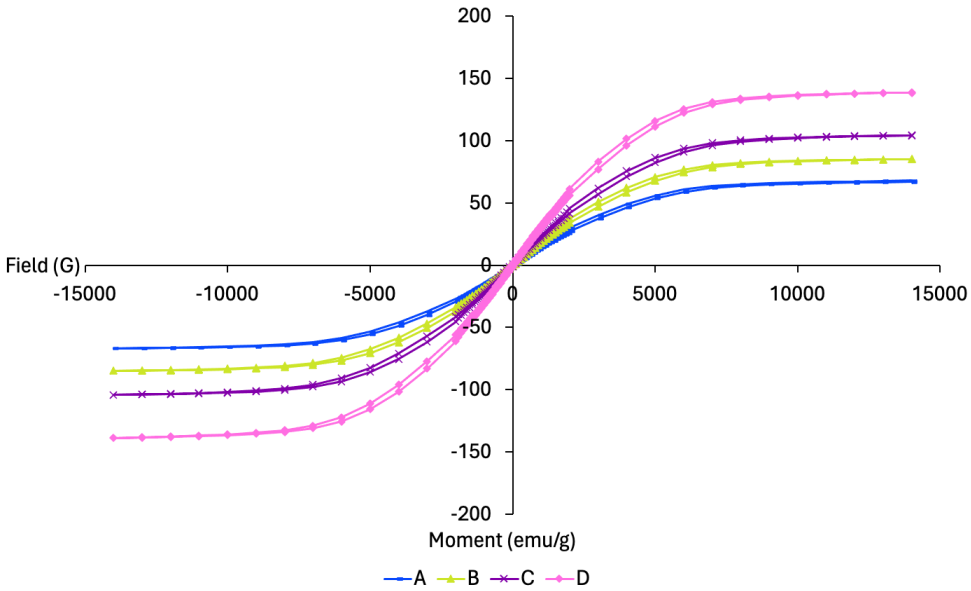


Fig. 2. Magnetization curve of the fabricated MR foam samples at room temperature in the range of -14 to 14 kA/m of magnetic field, where sample A is free foaming MR foam and sample B, C and D represents constraint volume foaming MR foam with 100%, 75% and 50% volume respectively

Generally, the value of magnetic saturation (M_s) for MR materials is determined by the content of magnetic particles in the material [27,30]. In this study, the result shows that MR foam fabricated under a free foaming condition, sample A, has the lowest M_s about 67.43 emu/g. Whereas, when 100% volume of constrained foaming was introduced as the fabrication process (sample B), the M_s showed an increment up to 26%, where the value rose up to about 84.99 emu/g as compared to sample A. Indeed, as the volume of constrained foaming was further reduced by 25% (sample C) and 50% (sample D), the M_s portrays another increment trend showing the value increases to about 104.12 emu/g and 138.68 emu/g, respectively. This indicates that the increment in M_s value was in accordance with the decreasing volume of constrained foaming as displayed in Figure 2. It was believed to happen due to the changes in the morphological structure of MR foam samples that certainly affect the distribution of CIPs in the foam structure [14]. As previously reported, the introduction of constraint volume of foaming process causes an enhanced in the pore development of MR foam structure hence a more continuous magnetic moment would be able to form when it was subjected to magnetic field [14]. Admittedly, it was reported that free foaming MR foam has large pore of foam structures that lead to random and farther particle distribution that caused restriction to the magnetic flux flow. For this reason, higher M_s can be observed in sample B than sample A, mainly due to a better foam structure and compact distribution of CIPs during the constrained foaming processes. Apparently, as the volume of mould was further reduced (sample C and D), higher M_s was observed. This explains that the CIPs distribution became more compact, reducing the gaps between particles in the foam structure, allowing the CIPs to swiftly magnetized and aligned with the direction of magnetic field hence leading to higher M_s values.

In addition, the value of coercivity (H_c) of the MR foam samples were also observed and summarized in Table 2. Generally, H_c is the intensity of magnetic field required to reduce the magnetization of the MR foam to zero after it has been magnetized. In essence, the H_c for all samples showed no distinct changes as the percentage difference between all samples is about 1%. This indicates that the CIPs still portray their own characteristic as soft magnetic particles without having been influenced by the matrix of MR foam. The value of H_c is about 45 G – 46 G which is in the range

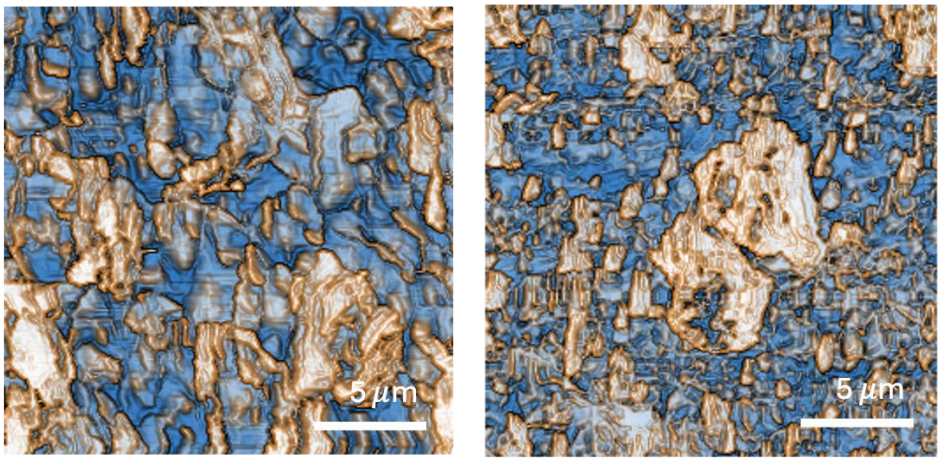
as reported by previous studies [22]. However, in detail observation, the value of H_c showed a small increasing trend then followed by a decreasing trend. Initially, the value of H_c increased from 45.829 G to 46.308 G when constraint volume foaming process was introduced as displayed by the results of sample A and B, respectively. Then, the value slightly increased to about 46.365 G as the volume of constrained foaming reduced to 75% from the 100% volume (sample C). As the volume was further reduced to 50% (sample D), the value of H_c portrayed a decrement to about 45.028 G. This is expected to happen due to the concentration of CIPs has reached the limits where the interactions might reduce the H_c due to increased interference among the CIPs. Depending on particle interactions, optimal concentration increases the MR foam resistance to demagnetization. Therefore, these properties are crucial to be observed since it is critical for the effective application of MR foam in its potential application such as in soft robotic applications where adjustable magnetic properties are essential. Thereupon, a high M_s would have the advantage of allowing the materials to instantly response towards the magnetic field, while low M_r and H_c making these MR foam able to repeatedly provide a response within a short time thus suitable for the suggested potential applications.

Table 2
Comparison of magnetic properties between MR foam fabricated by different foaming processes

Sample	Magnetization saturation, M_s (emu/g)	Percentage increase of M_s (%)	Coercivity, H_c (G)	Remanence, M_r (G)
A	67.4294	-	45.829	0.7234
B	84.9894	26	46.308	0.9235
C	104.1208	54	46.365	1.1109
D	138.6758	71	45.028	1.4565

3.2 Microstructure Analysis

To support this finding, the determination of MR foam surface characteristics of a free foaming and 50% volume of constrained foaming MR foam samples was analysed using AFM. This analysis is essential for characterizing the material in a localized area and to provide the correlation between the effect of constraint volume foaming process with the distribution of CIP particles embedded in the struts of the foam structures towards its magnetic properties. This analysis uses a type of probe-scanning microscope that has been the most influential techniques for characterizing materials in terms of phase separation especially for composite materials. It provides information through digital images which were generated from the deflection of the cantilever during the scanning process being conducted. The AFM topography evaluation of MR foam surface is thought to significantly influenced and reflect the bulk properties of MR foam since generally, the properties of porous materials are highly dependent to its structure itself [31]. Consequently, Figure 3 (a) and (b) provide images of microphase segregation of different phases, specifically CIPs and the polyurethane (PU) matrix, which were observed using tapping mode AFM analysis, for MR foam samples fabricated via free foaming (sample A) and 50% constrained volume of foaming process (sample D) accordingly. Figure 3 (a) and (b) presented the phase images of the strut structure within the scanning area of 20 µm × 20 µm for samples A and D, respectively. Basically, a rigid domains typically appear in a lighter color images, while softer domains appear in a darker color images [32-34]. Therefore, based on observation, the presence of lighter domains scattered around the scanning area is identified as CIPs which were in line with previous studies reported that CIPs was well distributed in the struts of the MR foam samples [11,13,14,16,19]. The variation in size suggests that CIPs were embedded at different depth within the strut structures. Following this, the darker color indicated the matrix of MR foam samples.



(a) (b)

Fig. 3. AFM images of (a) free foaming MR foam (sample A) and (b) 50% constrained foaming MR foam (sample D)

In the analyzed images, the lighter domains of sample A (Figure 3 (a)) appear to be more widely spaced compared to sample D (Figure 3 (b)), where the lighter domains are closer together and more frequently observed. This indicates a higher distribution and concentration of CIPs per unit volume in sample D, which directly explained and supported the result of highest M_s value of sample D as discussed in previous section. Indeed, this finding definitely agrees with previous finding where it was reported that higher storage modulus was observed in constraint volume foaming MR foams due to higher concentration and compact distribution of CIPs [14]. When the volume of MR foam was reduced to 50% from its original volume under a constrained foaming condition, the MR foam was expected to experience a restriction during the foaming process where it has limited space to expand during the nucleation of pores happened. As the results, smaller pores was observed and thicker struts were formed indicating more CIPs were accommodated per unit volume of the struts [14]. As mentioned earlier, the variation in the size of lighter domains is due to particles being embedded at different depths in the darker domains, which are the matrix. This is primarily due to the restricted expansion of sample D, resulting in a denser particle distribution. Generally, the reduced distance between CIPs enhances the formation of chain-like structures when MR foam is exposed to a magnetic field [35]. As the magnetic field was applied to the MR foam samples, CIPs align with the direction of the magnetic field, and a compact distribution improves particle interactions, leading to better properties of MR foams.

4. Conclusions

This study presented a novel approach to fabricate MR foam samples via different constraint volumes foaming processes. A free foaming MR foam (sample A) together with a 100%, 75% and 50% volume of constrained foaming MR foams (sample B, C and D, respectively) was fabricated in order to analyse the effect of different foaming process towards its magnetic properties. Overall, it can be observed that constraint volume foaming process has significantly improved the distribution of CIPs within the MR foam matrix, leading to an increase in the overall properties of the material. The magnetic properties analysis was conducted using VSM for the observation on the effect of constraint volume of foaming process towards the magnetic properties, while the morphological analysis using AFM was carried out to support the overall findings for this study. In conclusion, the enhancement of magnetic properties of MR foam can be attributed to the reduced distance between the CIPs which allows more connectivity of magnetic moment to form under the influence of magnetic field.

However, it was observed that different constraint volumes foaming process do not significantly impact the remanence and coercivity of the MR foam indicating the intrinsic magnetic behaviour of the CIPs remains unaffected by their distribution within the matrix. The AFM analysis provided clear evidence supporting these findings, showing that CIPs in free foaming MR foam samples (sample A) are farther apart compared to 50% volume of constrained foaming (sample D). Due to their synergistic behavior, it can be concluded that MR foam will have the potential to be further investigated in the near future to develop more advanced multifunctional devices.

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