A New Magnetorheological Composite Gel and Its Controllable Rheological Behaviour

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Abstract: A series of magnetorheological composite gels (MRCG) were prepared using carbonyl iron powder as the magnetic particles, a complex of methyl silicone oil and gelatin/agaropectin colloids as the matrix and various additives. The rheological behaviour of the samples was tested using a rheometer in rotary and oscillatory shear modes. The results demonstrate that rheological behaviour is significantly dependent on the colloid content of the matrix. The shear yield strength increases with increasing colloid content of the matrix and reaches a maximum of 92.1 kPa. The linear viscoelastic range of the MRCG is almost independent of colloid content. In addition, the shear storage modulus of the MRCG increases with increasing colloid content, and the maximum value of the change in absolute modulus is 2.526 MPa. Adding nanosized SiO_2 can significantly improve the shear yield stress and broaden the controllable range of the MRCG under a magnetic field.

Keywords: Magnetorheological composite gels, Rheological behaviour, Colloid content, Shear storage modulus, Nanosized SiO_2.

1. INTRODUCTION

A magnetorheological gel (MRG) is prepared by uniformly dispersing micron-sized magnetic particles in a polymer gel matrix to form a stable, gelatinous system in which the mechanical properties can change rapidly under an external magnetic field. The MRG not only retains the excellent magnetorheological effect of a magnetorheological fluid (MRF) [1] but also significantly improves sedimentary stability compared with that of MRF. The concept of the MRG was first proposed by Shiga *et al*. [2] in 1995, and this kind of semi-solid system, which has properties between those of the MRF and magnetorheological elastomer (MRE), has become an important branch of the research of magnetorheological materials. Parts of the polymer gels are adsorbed on the surfaces of magnetic particles, and the formation of reticular structure increases the distance between particles, weakening the Van der Waals force, reducing the aggregation and enhancing the dispersibility of particles. Therefore, many scholars have prepared many varieties of MRGs with different kinds of polymer gel systems: Wei *et al*. [3] developed an MRG based on the polyurethane matrix and studied the rheological behaviour under static and dynamic shear conditions; Park *et al*. [4] prepared a novel kind of MRG by uniformly dispersing carbonyl iron (CI) microspheres in grease and studied the rheological characteristics; Mitsumata *et al*. [5] examined the influence of carrageenan concentration on the rheological behaviour of carrageenan-based MRG; Kim *et al*. [6] prepared high-performance MRG by using two compounds, polyisobutene and polybutylene, as matrix and studied the rheological and sedimentary behaviours; Zhou *et al*. [7] designed an MRG with high shear yield stress through using the reactants, hexamethyldisiloxane and polyborodiphenylsiloxane, as carrier; and Qin *et al*. [8] studied the influence of iron powder content on the rheological behaviour of gelatin-based MRG. MRG is classified as either liquid-like or solid-like according to the state of the matrix. Liquid-like MRG, which is regarded as an enhanced version of MRF, has higher shear yield strength than MRF and great sedimentary stability of particles [9-10]. Properties of solid-like MRG are similar to those of MRE. The magnetic particles can form chains and other regular structures under a magnetic field before the matrix solidifies, and these regular structures remain fixed in the gel matrix after the magnetic field disappears [11-12].

There are many kinds of polymer gels, and their mechanical and thermal properties are different [13-14]. In addition to selecting various colloids as MRG matrices to study their performance by magnetic control, some new properties still need to be developed. Studies have shown that the transition of MRG from a liquid-like to solid-like state can be achieved by adding polymer gels to a low-viscosity liquid to change the content of polymer gels [15-18]. These studies also indicated that in some application fields, such as magnetorheological dampers and isolators, and other fields of vibration control [19-23], MRG exhibits more application potential than MRF.

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Polymer gels do not usually combine well with low-viscosity liquids, and the stratification is clear in the related studies above. Consequently, the shear yield strength of MRG is influenced, the choice of materials is complicated, and the cost is high. Therefore, this study focused on the preparation of novel magnetorheological composite gels (MRCG) with high composite performance and higher shear yield strength by using gelatin, agaropectin, methyl silicone oil and other common materials as matrix. In addition, the rheological behaviour was also studied.

2. EXPERIMENTAL PROCESS

2.1. Preparation of MRCG

CI powder with an average size of 3.5 μm was obtained from Jiangsu Tianyi Ultra-fine Metal Powder Co., Ltd., and was mainly used in the experiment; the specific performance is outlined in Table **1**. Gelatin (Chemical Pure, Shanghai Zhanyun Chemical Co., Ltd.), agaropectin (Biochemical Reagent, Guangzhou Saiguo Biotech Co., Ltd.), methyl silicone oil (Jiangsu Lvxun Chemical Co., Ltd.), and glycerol (Analytical Reagent, Sinopharm Chemical Reagent Co., Ltd.) were used as the matrix materials. Polyethylene glycol PEG-400 (Analytical Reagent, Tianjin Zhiyuan Reagent Co., Ltd.) and oleic acid (Analytical Reagent, Sinopharm Chemical Reagent Co., Ltd.) were purchased as the active agents. Absolute ethyl alcohol (Analytical Reagent, Tianjin BASF Chemical Co., Ltd.) was used as dispersant. A thixotropic agent and nanosized SiO_2 (50±5 nm, Chemical Pure, Shanghai Jingchun Biochemical Technology Co., Ltd.) in nano-thixotropic agent were also used in the experiment.

MRCG is a colloidal suspension system in which magnetic particles are uniformly dispersed in the carrier to have excellent working performance. Therefore, dispersing particles in the carrier was a crucial technical step during the preparation of the MRCG. The hydrophilic groups of the surfactant applied in the magnetorheological material were adsorbed on the surfaces of magnetic particles, and the lipophilic groups were easily dispersed in the carrier so that the magnetic particles can be better scattered in the carrier. At the same time, the surface activity depended significantly on the hydrophilic-lipophilic balance (HLB) of the surfactant. A higher HLB value demonstrates stronger hydrophilicity, and a smaller value indicates stronger lipophilicity; generally, the value of HLB is between 1 and 40. The values of HLB are important references in practical application. The HLB of a lipophilic surfactant is smaller, and that of a hydrophilic surfactant is higher. The turning point between hydrophilicity and lipophilicity is when the value of HLB is equal to 10. The surface activity is lipophilic when the value is less than 10 and hydrophilic when the value is more than 10. Two kinds of common surfactants, polyethylene glycol and oleic acid, were used to improve the properties of magnetic particles in the experiment. The HLB of polyethylene glycol was 20, which suggested that hydrophilicity was strong; the HLB of oleic acid was 1, which indicated that lipophilicity was strong. Research has shown that using these two surfactants can efficiently improve the shear yield strength and stability of an MRF.

The processing step of the CI powder and surfactant is as follows: (1) CI powder was placed into an absolute ethyl alcohol solution containing the surfactant, and the materials were placed into a ball mill jar; (2) A DW-2-80 W model electric stirrer was used to stir the materials in the ball mill jar for 10 min at 250 r/min; (3) Large and small steel balls with quantity scale values of 1 to 5 were placed in the ball mill jar, and the jars were placed in pairs into an M-3SP2 model double planetary mill for 30 min at 200 r/min; (4) The ball mill jars containing the liquid mixture were placed into a DZF-6020 model vacuum drying oven at 85℃ under a vacuum pressure of -0.09 MPa and dried for 480 min to remove the absolute ethyl alcohol. The modified iron powder was obtained after cooling in vacuum.

The preparation process is shown in Figure **1**. The specific preparation procedures of the MRCG are demonstrated as follows: (1) An electronic scale was used to quantify the gelatin and agaropectin powders. Water, glycerol and powders were heated and stirred in

Table 1: The Major Performance Indices of Carbonyl Iron Powder

Fe Content	C Content	O Content	N Content	Average Particle Size	Apparent Density	Tap Density
98.030%	0.730%	0.276%	0.964%	3.500 μm	2.800 $g{\cdot}cm^{-3}$	4.200 $g{\cdot}cm^{-3}$

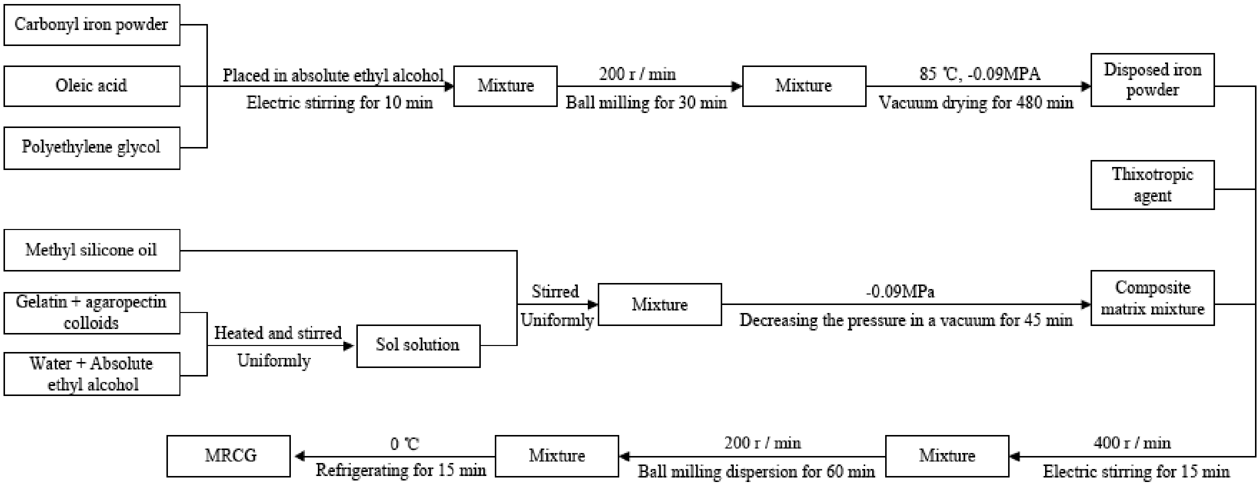

Figure 1: The preparation process of the MRCG.

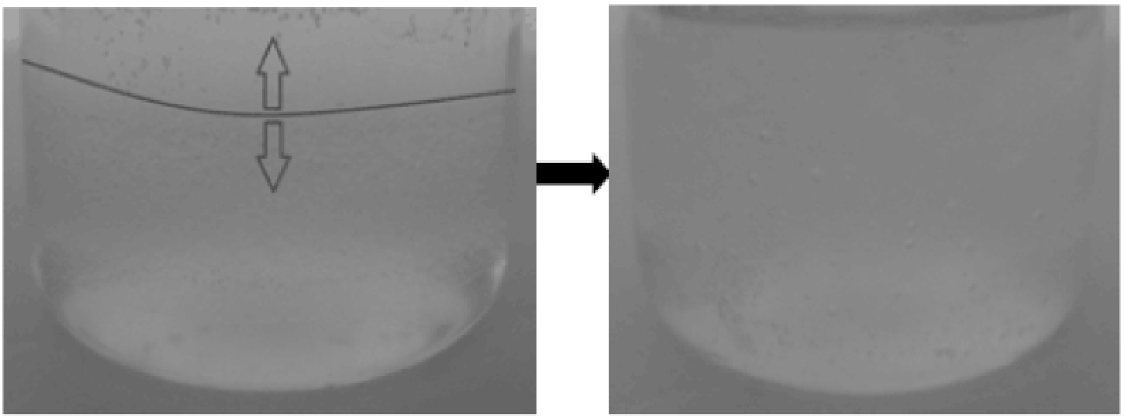

Figure 2: The matrix of MRCG, which consists of methyl silicone oil and gelatin/agaropectin colloids.

the same beaker until the powders were completely dissolved to form the colloidal solution. As shown in Figure **2**, stratification is clearly visible after adding quantitative methyl silicone oil, stirring uniformly and leaving the solution for a while. The level of the methyl silicone oil is above the marked line, and the colloidal solution is below it; (2) The beaker was placed in the vacuum drying oven and decompressed for 45 min at a vacuum pressure of -0.09 MPa to form a composite colloid (Figure **2**); (3) The quantitative matrix material was added to the ball mill jar. The modified CI powder and thixotropic agent were also placed in the jar in batches to be stirred using the electric stirrer for 15 min at 400 r/min. A B25 model high-shear emulsifying machine was also used to disperse the mixture at high speed; (4) Large and small steel balls with quantity scale values of 1 to 5 were placed into the ball mill jars, and the jars were placed in pairs into a double planetary mill for scatter at 200 r/min for 60 min; (5) The MRCG sample was prepared after pouring the magnetorheological colloid into a plastic flask of 100 mL, labelling and placing in a 0℃ refrigerator for 15 min.

Five groups of MRCG samples with good dispersibility were prepared, and the mass fraction of the CI powder was approximately 72%. The major components are demonstrated in Table **2**, and the dosage ratio of gelatin to agaropectin is 3 to 1.

2.2. Sample Characterization

A D8 Advance X-ray diffractometer from Bruker Co., Germany, was used to analyse the phase of the CI powder before and after processing with the active agent. The MRCG samples were tested in rotary and oscillatory shear modes using a rheometer from the MCR series (Anton Paar Co., Austria).

Table 2: The Mass Fractions of Major Components of MRCG Samples

Samples	Carbonyl Iron Powder	Matrix-25%	Thixotropic Agent
MRCG-1	72%	Silicone oil/Matrix-100% Colloids/Matrix-0%	3%
MRCG-2	72%	Silicone oil/Matrix-90% Colloids/Matrix-10%	3%
MRCG-3	72%	Silicone oil/Matrix-80% Colloids/Matrix-20%	3%
MRCG-4	72%	Silicone oil/Matrix-70% Colloids/Matrix-30%	3%
MRCG-5	72%	Silicone oil/Matrix-60% Colloids/Matrix-40%	3%

3. RESULTS AND DISCUSSION

3.1. Phase Analysis

XRD spectra of the CI powder before and after processing are shown in Figure **3**. As shown in the figure, the CI powder exhibited strong diffraction peaks at 2θ=40.4°, 45°and 82.6° both before and after processing, which are consistent with the standard spectra of Fe. Only the peak intensity changes and the peak intensity of the CI powder after processing decreased slightly, which may be related to the fact that a layer of active agent was absorbed on the surfaces of the particles and the crystal structure of Fe exhibits no change after processing.

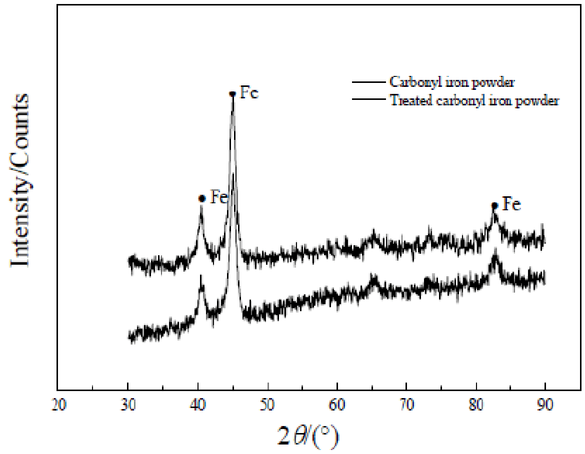

Figure 3: XRD spectra of the CI powder before and after processing.

3.2. Rotary Shear Test

Figure **4** shows the shear yield stress with changing magnetic flux density and the shear yield strength with changing colloid content of the matrix. It is observed that the shear yield stress of the MRCG greatly depended on the magnetic flux density and colloid content of the matrix when the shear rate is 100 s^{-1}. As shown in Figure **4**(**a**), the shear stress of the MRCG increases with increasing magnetic flux density until magnetic saturation. When the magnetic flux density increases gradually from 0 to 0.4 T, the shear stress of the MRCG becomes nearly independent of the colloid content of the matrix. When the magnetic flux density is more than 0.4 T, the shear stress of the MRCG greatly depends on the colloid content of the matrix: the higher the colloid content is, the higher the shear yield strength. MRCG-1 without any colloid, known as MRF, exhibits the lowest shear yield strength in Figure **4**(**b**) at 65.9 kPa. With the continuous addition of colloids, the shear yield strength can reach 92.1 kPa when the colloid content is 40 wt%. Consequently, shear yield strength is increased by adding colloids to the MRF, and the formed MRCG is a liquid-like colloid. The shear yield stress under a magnetic field increases with increasing colloid content, and the magnetically induced shear yield stress is 90.9 kPa when the colloid content is 40 wt%.

Figure **5** and **6** show the changes in shear stress and apparent viscosity of MRCG-2 with shear rate under different magnetic flux densities. As shown in Figure **5**, the shear stress of the MRCG increases with increasing shearing rate; additionally, the higher the magnetic flux density is, the larger the shear stress. That is because the higher magnetic flux density represents a larger magnetic force between the particles and a more ordered microstructure inside the MRCG. Figure **6** shows that the apparent viscosity of the MRCG decreases with increasing shearing rate, exhibiting the typical non-Newtonian fluid characteristic known as the shear-thinning phenomenon, which indicates that the liquid-like MRCG is a pseudo plastic fluid [24].

Figure **7** shows the changes in apparent viscosity of the MRCG with magnetic flux density. One can observe that the MRCG clearly exhibits the magneto-viscous effect (MVE) in which the apparent viscosity of the MGCR increases significantly with increasing external

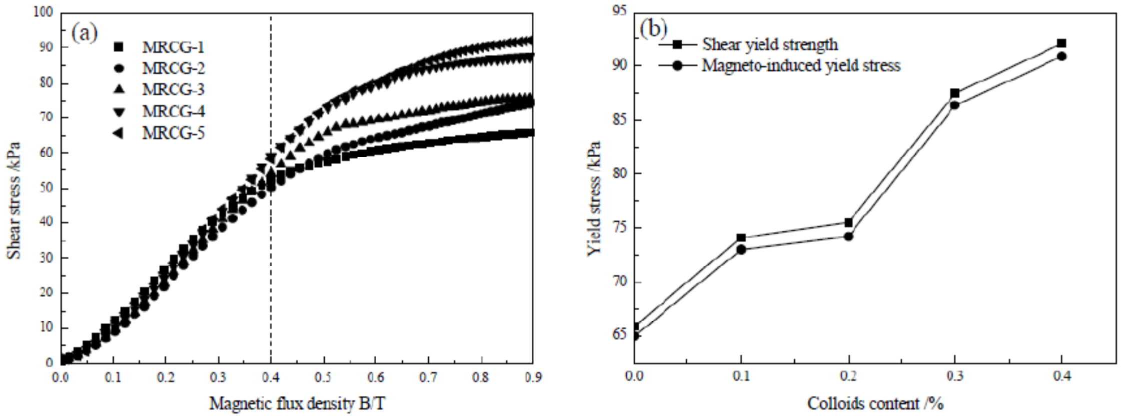

Figure 4: The relationship between (**a**) shear yield stress and magnetic flux density and (**b**) shear yield strength and colloid content of MRCG (shear rate of 100 s^{-1}).

magnetic field [25]; in addition, the higher the colloid content is, the greater the change in apparent viscosity.

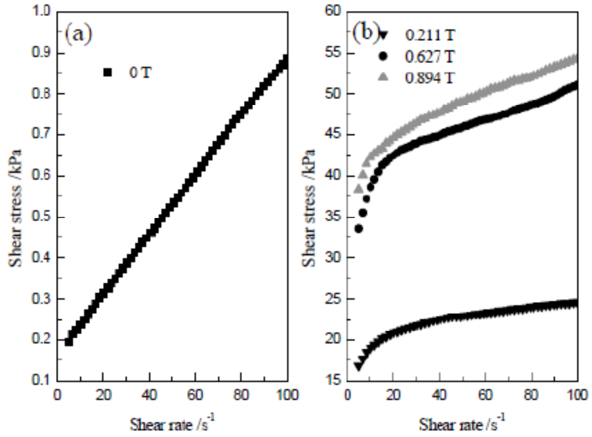

Figure 5: The relationship between shear stress and shear rate of MRCG-2 (**a**) under no magnetic field and (**2**) under different magnetic flux densities.

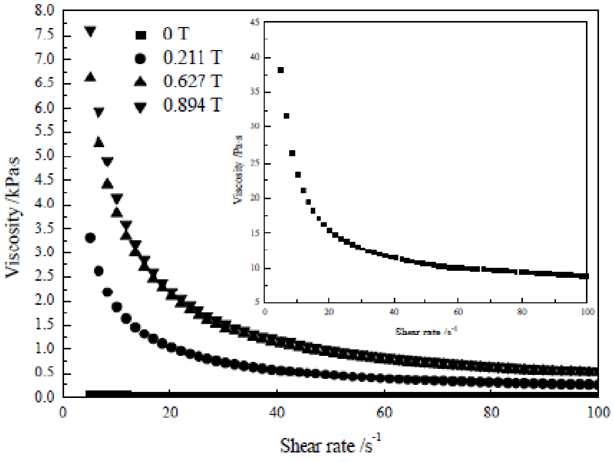

Figure 6: The relationship between apparent viscosity and shear rate of MRCG-2 under different flux densities.

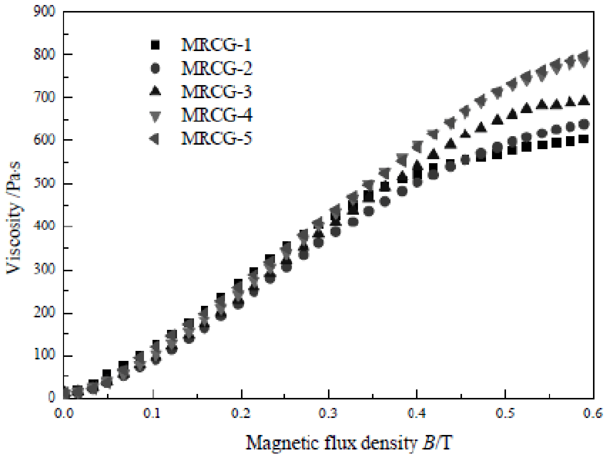

Figure 7: The changes in apparent viscosity with magnetic flux density of the MRCG.

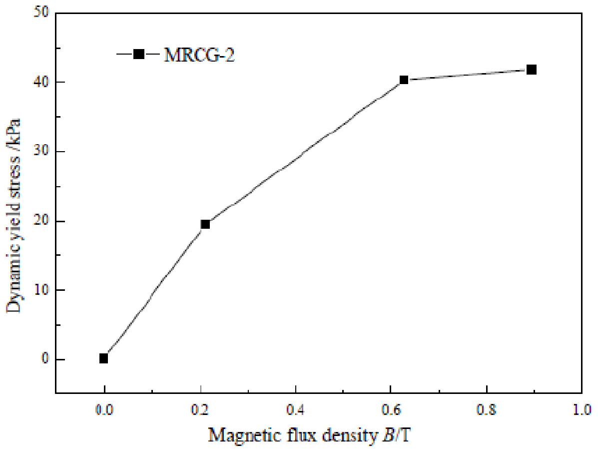

Figure 8: The relationship between dynamic shear yield stress and magnetic flux density.

The trends shown in Figures **4** to **7** indicate that rheological behaviour can be controlled not only by regulating the magnetic field but also by changing the colloid content. The rheological and shear-thinning phenomena can be demonstrated by a non-linear model:

$$\tau = \tau_y + K\dot{\gamma}(1 - \mu\dot{\gamma}) \tag{1}$$

where τ denotes the shear stress of the MRCG, $\dot{\gamma}$ denotes the shear rate, τ_y is the dynamic shear yield stress depending on the magnetic field and μ indicates the degree of shear-thinning of the MRCG. The model was used to fit the curves in Figure **5** to obtain the value of τ_y, and the fitting process was performed for the curves at the shear rate of 20 s^{-1}. The relationship between the dynamic shear yield stress of MRCG-2 and magnetic flux density is shown in Figure **8**.

Figure **8** indicates that the dynamic shear yield stress mainly depends on the magnetic field intensity. τ_y increases with increasing magnetic field intensity; furthermore, values of τ of MRCG in rotary shear mode increases significantly under a magnetic force. The larger the magnetic field is, the stronger the magnetic force between the particles and the more ordered the interior structure of the MRCG [26].

3.3. Oscillatory Shear Test

The linear viscoelastic range, LVE, is crucial to viscoelastic materials. The dynamic mechanical property of a viscoelastic material has special physical meaning within this range in that the destruction of the microstructure of the material by an external excitation signal is negligible and the destruction of the microstructure by strain is not considered if the excitation strain amplitude is set within the LVE [27-29]. The LVE of the MRCG with different colloid contents is easily obtained by strain amplitude scans using the rheometer in oscillatory mode.

The relation curve between shear storage modulus and strain amplitude of the MRCG is shown in Figure **9** for a magnetic flux density of 0.894 T and an oscillation frequency of 10 Hz. The figure demonstrates that the LVE of the MRCG is almost independent of the colloid content of the matrix and that the values of LVE of the MRCG with 10%, 20%, 30% and 40% (wt) colloids range from 0 to approximately 0.1%. Therefore, the higher the colloid content is, the larger the shear storage modulus of the MRCG.

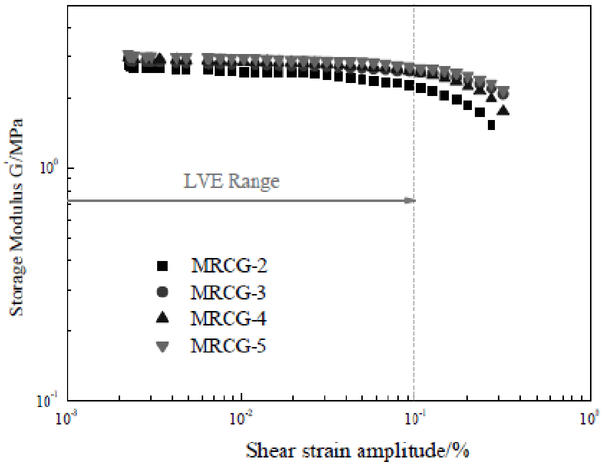

Figure 9: The relationship between storage modulus and shear strain amplitude of the MRCG (B=0.894 T).

The relation curves in Figure **10** show changes in shear storage modulus and loss factor of the MRCG with magnetic flux density. The shear storage modulus of the MRCG increases with increasing magnetic flux density, and the loss factor decreases with magnetic flux density. The dynamic mechanical property of the MRCG greatly depends on the colloid content of the matrix; the higher the colloid content is, the larger the storage modulus; however, the loss factor is smaller. The specific parameters of various samples with added colloids are shown in Table **3**, which indicates that the higher the colloid content is, the larger the initial shear storage modulus, the higher the changes in absolute modulus and the smaller the initial loss factor. Table **3** demonstrates that the MRCG is transformed from a liquid-like to solid-like state with increasing colloid content.

Different mass fractions of nanosized SiO_2 were added, and the mass fractions of the CI powder and thixotropic agent were used as constants to study the

Table 3: The Major Dynamic Mechanical Property Parameters of the MRCG

Name of sample	MRCG-2	MRCG-3	MRCG-4	MRCG-5
Initial modulus G_0'/ MPa	0.0092	0.0151	0.0162	0.1040
Changes in absolute modulus $\Delta G'$/ MPa	1.7110	2.3750	2.4840	2.5260
Initial loss factor $\tan\delta_0$	0.6560	0.6430	0.6240	0.4730

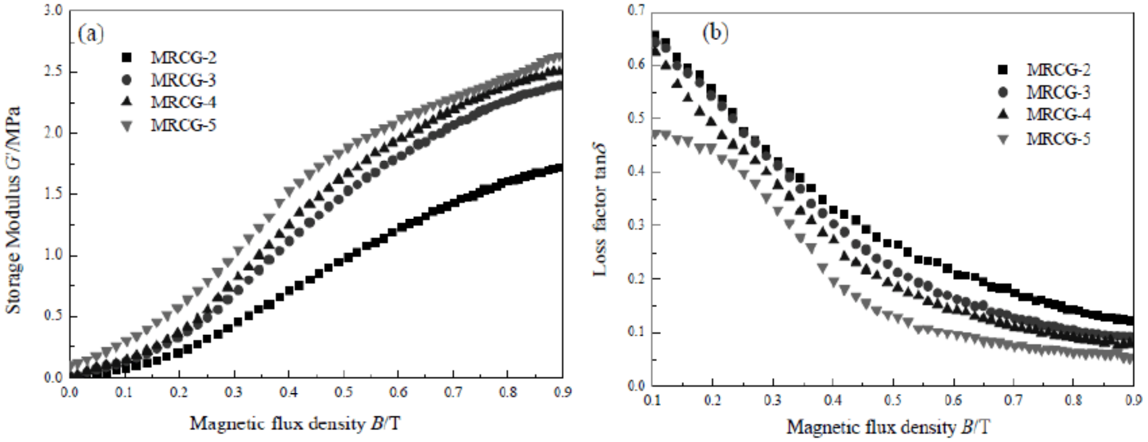

Figure 10: The relationship between magnetic flux density and (**a**) shear storage modulus and (**b**) loss factor of the MRCGs (strain amplitude is set to 0.1%).

influence of nanosized SiO_2 on the shear stress of the MRCG on the basis of the formula of MRCG-2. The results are demonstrated in Figure **11**.

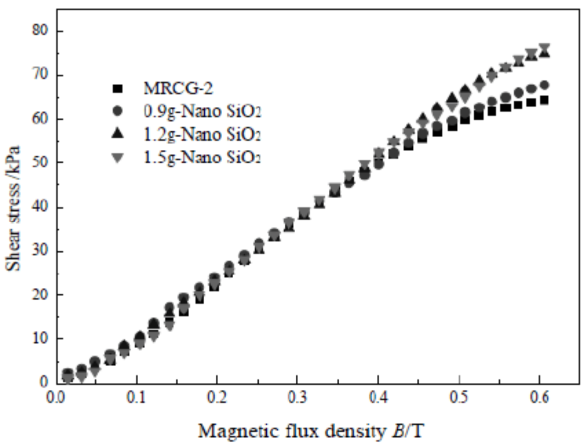

Figure 11: The relationship between shear stress of the MRCG and different quantities of nanosized SiO_2 and magnetic flux density.

Figure **11** indicates that adding nanosized SiO_2 can improve the shear yield stress of the MRCG. The shear yield stress of MRCG-2 without nanosized SiO_2 is 64.5 kPa when the magnetic flux density is 0.6 T. The shear yield stress of samples with 0.9 g, 1.2 g and 1.5 g (mass fractions of 0.75%, 1% and 1.25%, respectively) nanosized SiO_2 reached 67.8 kPa, 74.9 kPa and 76.6 kPa, respectively. The specific parameters are shown in Table **4**.

Table 4: The Major Parameters of MRCG Samples with Different Quantities of Nanosized SiO_2

Quantity of SiO_2 added (g)	0	0.9	1.2	1.5
Shear stress under 0.6 T/ kPa	64.5	67.8	74.9	76.5
Magnetically induced shear stress/kPa	63.2	65.4	73.2	75.3

The variation range of shear stress can be broadened with nanosized SiO_2 when the magnetic flux density changes from 0 to 0.6 T. That is because the interior magnetic particles of the MRCG form chain-type and other structures under the magnetic field, and a structural space with a certain gap is formed through the interactions between chains. The nanosized SiO_2 is dispersed in the structural gap along with the colloids during the chain-forming process to accommodate the structural weakness. As a result, when the solidified MRCG is sheared, additional energy is needed to destroy the structure.

CONCLUSIONS

1. The shear yield strength of the MRCG with 40 wt% colloids in the matrix reaches 92.1 kPa, and the strength increases by 26.2 kPa, compared with those of the MRCG without any colloids. The shear yield strength of the MRCG and the magnetically induced shear yield stress increase with increasing colloid content, and the magnetically induced shear yield stress reaches 90.9 kPa when the colloid content is 40 wt%. Meanwhile, the rheological and shear-thinning behaviour of the MRCG can be described by a non-linear model.

2. The LVE of the MRCG is almost independent of colloid content, and the value is approximately less than 0.1%. The storage modulus of the MRCG increases with increasing magnetic flux

density; the higher the colloid content is, the larger the storage modulus of the MRCG and the larger the change in absolute modulus. The change in absolute modulus reaches a maximum of 2.526 MPa when the colloid content is 40 wt%. However, the loss factor of the MRCG decreases with increasing magnetic flux density; the higher the colloid content is, the smaller the loss factor, which demonstrates that increasing the magnetic field leads to the transition from a liquid to solid rheological behaviour of the MRCG and that adding colloids continuously leads to the transition from a liquid-like to solid-like state of the MRCG.

3. The shear yield stress and magnetically induced shear stress of the MRCG can be increased by adding nanosized SiO_2 to the MRCG. The shear stress and magnetically induced shear stress of the MRCG with 1.5 g SiO_2 under 0.6 T are 76.5 kPa and 75.3 kPa, respectively, and increase by 12 kPa and 12.1 kPa, respectively, compared with those of the MRCG without nanosized SiO_2. The higher magnetically induced shear stress indicates a larger adjustable range for the MRCG under a magnetic flux, and the range can be broadened by the addition of nanosized SiO_2.

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