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Fabrication and electro-responsive electrorheological characteristics of rice husk-based nanosilica suspension

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ABSTRACT

Rice husk-based nano-sized silica particles were prepared using both acid and thermal treatment methods for applications to electro-responsive electrorheological (ER) fluids when dispersed in silicone oil. The morphology of the nano-sized silica particles was observed by scanning electron microscopy, and their chemical structure and thermal stability were examined by Fourier-transform infrared spectroscope and thermo-gravimetric analysis, respectively. The ER properties of the nano-sized silica particle-based ER suspension was investigated using a rotation rheometer under various electric field strengths, showing a conduction mechanism with a slope of 1.5 for a plot of the yield stress versus the applied electric field strength. In addition, its shear stresses increased with increased electric field strengths along with ER efficiency and became stable over the entire shear rate range when the electric field reached 2.0 kV/mm. Typical solid-like behaviors of the ER suspension was also confirmed by both higher storage moduli than the loss moduli and shear relaxation modulus property.

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1. Introduction

Electro-responsive electrorheological (ER) fluids have attracted considerable attention because of their interesting smart characteristics and applications under electric fields [1–4]. ER fluids are suspensions of dielectric and/or semi-conducting particles dispersed in a non-conducting liquid medium. Silicone oil is the most frequently adopted suspending medium for the fabrication of ER fluids with its various advantages such as low dielectric constant, optical transparency, chemical inertness, non-toxicity, nonflammability, and low volatility possessing wide range of kinetic viscosity [5]. Many types of dielectric particles ranging from synthesized inorganic particles [6,7] to core-shell particles [8–14] and hollow particles [15,16] have been used successfully for ER fluids.

The application of an external electric field to ER fluids results in a phase-transition from liquid-like to solid-like as a result of particle polarization [17]. At this final state, the dielectric particles are polarized into both negatively and positively charged portions at each end, resulting in the formation of chain particles owing

to their rapid electrostatic interactions within a few to dozen milliseconds [18]. The resulting response is reversible, meaning that when the external electric field is removed, they will change from solid-like to liquid-like immediately. Accordingly, ER fluids have been introduced in various engineering fields, such as shock absorbers, robotics, drug delivery, microfluidics, and tactile displays [19–22].

Although the synthesized electro-responsive particles are considered the most common dispersed phase for fabricating ER fluids, the trend in using bio-particles for ER fluids is being increased due to the environmental advantages. In the present study, rice husk-based silica nanoparticles were assessed for their potential applications to ER fluids.

Silica is one of the most general materials in the planet and can either be found in sand and quartz or be synthesized *via* chemical reactions. Silica can also be extracted from rice husk, a by-product of rice farming, because during their growth, the rice plants require more chemical elements, including silicon, which also plays many important roles in rice plant growth, such as developing resistance to specific stresses and disease [23]. In addition, silica helps minimize the toxicity of the minerals, including iron and aluminium, while increasing the uptake of phosphorus (P), further increasing tolerance to drought along with salt resistance by forming the silicified tissue in the plants. Meanwhile, as for a by-product of rice mill processing, most of the rice husk is either burned or discarded

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into the environment, causing both air and land pollution. Therefore, the use of rice husk as the source of silica production will enhance the value of this agricultural by-product.

Silica particles have been studied extensively for ER fluids, either in their pristine form due to adsorbed moisture on their surface or as silica composite particles coated with electro-responsive conducting materials. Initially, both pure silica and surface-modified silica have been introduced [24,25]. Silica particles coated with various conducting particles were studied further to improve the ER property [26], such as core-shell structured polypyrrole (PPy)-coated silica particles [27]. These also include monodispersed copolymer/silica core-shell nanoparticles [28] and graphene oxide-coated silica particles, exhibiting improved ER property [29].

In this study, silica nanoparticles were fabricated from rice husk using both acid and thermal treatment methods. The fabricated bio-silica nanoparticles were then used as a dispersed phase in ER fluids. Various characteristics of both silica and bio-silica-based ER fluids were examined, including the chemical structure by Fourier-transform infrared (FT-IR) spectroscopy, thermal properties by thermogravimetric analysis (TGA), the morphology by scanning electron microscopy (SEM), and the ER properties under different external electric field strengths.

2. Experimental

2.1. Materials and synthesis of nano-sized silica particles

Rice husks were collected from Huu Duc Food Commerce Co., Vietnam. H_2SO_4 was purchased from Xilong Scientific Co., Ltd (China). The rice husk after collection from rice mill processing were washed several times with water to remove dust and some unnecessary components. The rice husks were dried using an oven at 50 °C for one day before treatment with the acid solution. About 100 g of the dried rice husk was immersed in 400 ml a solution of 10 wt% H_2SO_4 and heated to 80 °C for 3 h. Subsequently, the rice husk was cleaned repeatedly using the distilled water by reaching $\text{pH} = 7$. The rice husk was dried again in an oven at 50 °C for 3 h. In the final step, the dried rice husk was heated in a furnace oven at 800 °C for 4 h to afford a white powder. The H_2SO_4 solution played a vital role in removal of the impurities existing in the rice husk and prevented the formation of black particles [30]. Beside it, the sulfuric solution also helped to accelerate hydrolysis and decomposition of organic components at a high temperature (800 °C) such as cellulose, hemicellulose and etc. [31]. The weight percent of bio-silica determined by being weighted again using an analytical balance was about 13 wt%.

2.2. Preparation of ER fluid

A sample of 10 vol% nano-sized silica particles was dispersed in silicone oil (kinematic viscosity: 50 cSt) to form an ER fluid. The fluid was then shaken and sonicated to promote dispersion.

2.3. Characterization

The chemical structure and thermal properties of rice husk-based silica produced were examined by FT-IR and TGA, while the morphology and element analysis of the nano-sized silica particles were examined by SEM (S-4300, Hitachi, Japan) with an energy-dispersive X-ray spectroscopy (EDS) (Horiba, Japan) accessory after platinum (Pt) sputtering. A pycnometer (Accupyc 1330, USA) was adopted to test density of the fabricated particles, giving 2.8 g/cm^3 . Electrical conductivity of the nano-sized silica particles was measured to be $8.1 \times 10^{-8} \text{ S/cm}$ using a resistivity meter (MCP-T610, Mitsubishi, Japan).

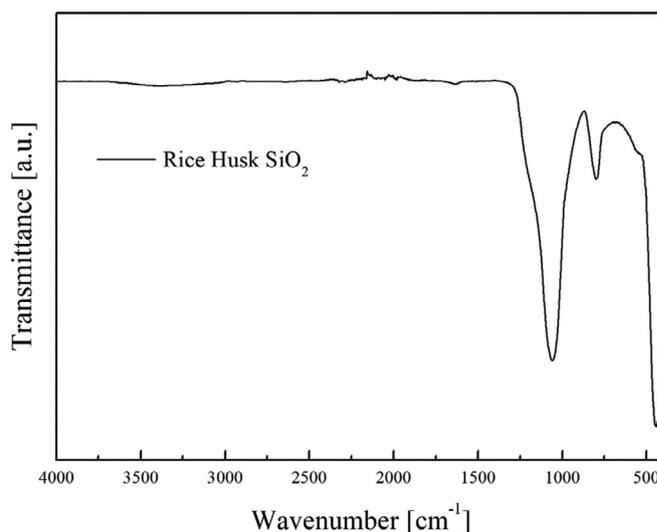


Fig. 1. FT-IR spectra of nano-sized silica.

For the ER tests, initially optical microscopy (OM) (BX51, Olympus, U.S.A.) attached with a high voltage generator was used to observe the chain-like structure of the nano-sized silica particle-based ER fluid directly. The ER behavior of the nano-sized silica-based ER fluid was examined by a rotation-typed rheometer (MCR 300, Anton Paar, Austria). Both a high d.c. voltage generator and a concentric cylinder geometry (CC17) were attached to the rheometer.

3. Results and discussion

The fabricated rice husk-based silica was white in the powder state and its chemical structure was confirmed by FT-IR spectroscopy. The absorption peaks at 1056 cm^{-1} , 804 cm^{-1} and 474 cm^{-1} in Fig. 1 were characteristic of silica particles. The absorption peaks at 1056 cm^{-1} and 804 cm^{-1} were designated to anti-symmetric and symmetric stretching vibration of Si–O–Si, respectively. The absorption peak at 474 cm^{-1} is related to bending vibration of Si–O–Si bond, confirming the existence of silica.

SEM showed that the rice husk-based nano-sized silica particles had irregular shapes, as shown in Fig. 2(a) and (b). The SEM image also showed their size in the range from 80 to 100 nm. In addition, we adopted the EDS analysis at a large surface area of the SEM mapping image to analyze the element composition of the nano-sized silica particles. Fig. 2(c) shows the EDS analysis result of the rice husk-based nano-sized silica particles, demonstrating element compositions of the nano-sized silica, such as silicon and oxygen. This confirmed that the synthesis of rice husk based nano-sized silica was successful.

Fig. 3 presents TGA curves of the rice husk-based silica. In Fig. 3, the initial weight loss due to the extraction of water molecules and dehydration of the residual silanol groups [32] was observed. After 400 °C, the next weight loss was designated to the decomposition of functional group containing oxygen on silica surface. The amount of weight loss of silica was only 6.9% at 800 °C.

The ER phenomenon of the nano-sized silica particle-based ER fluid was conducted by OM attached to a high voltage source. In absence of an applied electric field (Fig. 4(a)), the nano-sized silica particles were dispersed randomly in the silicone oil. By introducing a voltage (Fig. 4(b)), the nano-sized silica particles began to move toward the electrodes and formed chains between the electrodes, exhibiting an ER fibril structure under the applied electrical field.

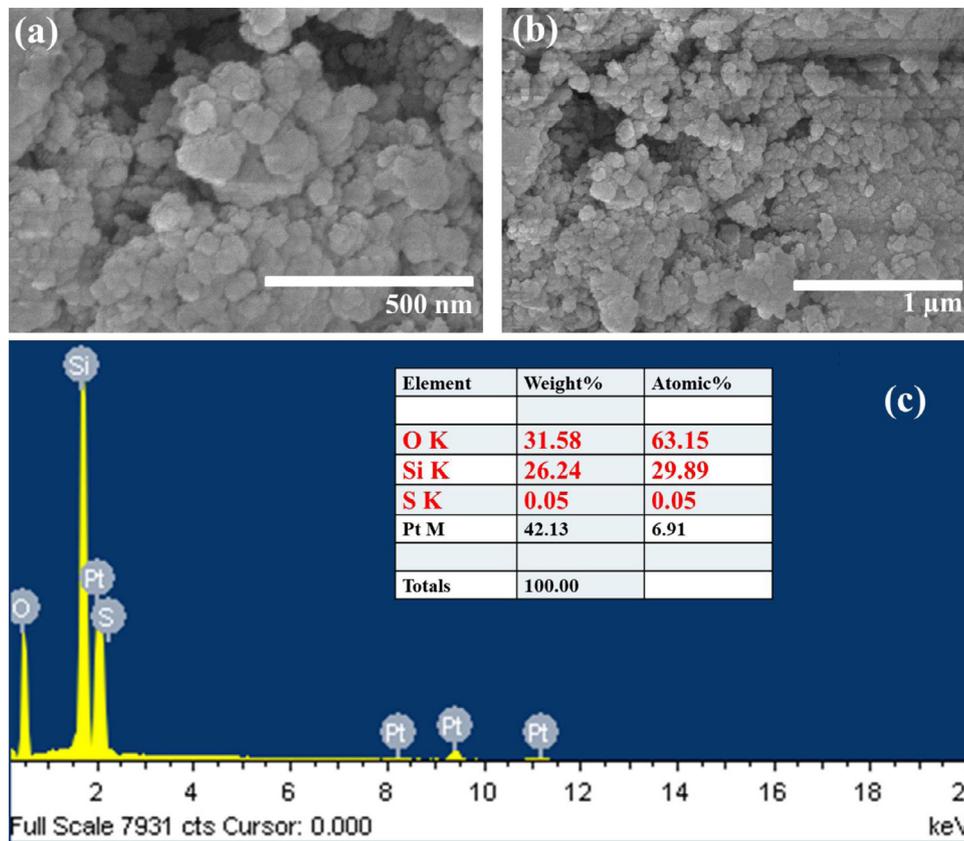


Fig. 2. SEM image (a) and (b) and EDS data (c) of nano-sized silica particles.

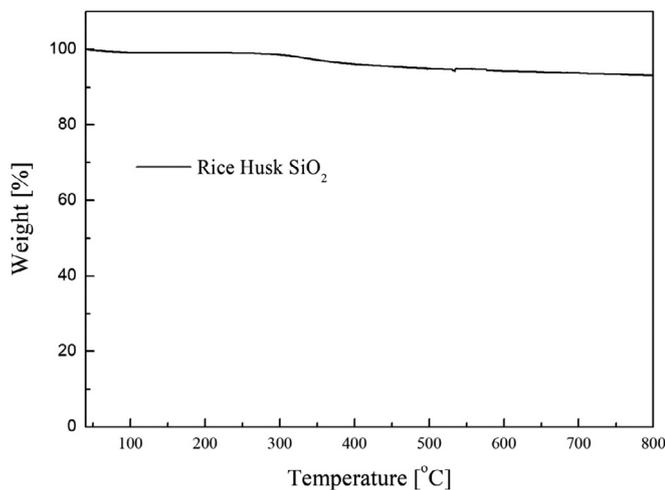


Fig. 3. TGA curve of nano-sized silica.

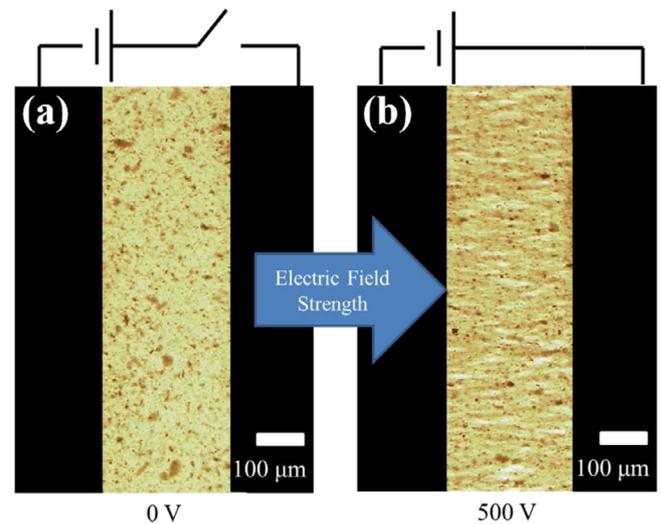


Fig. 4. OM images of the nano-sized silica particles without (a) and with (b) an electric field strength (500 V).

Fig. 5 shows (a) shear stress and (b) viscosity of the nano-sized silica-based ER fluid with a 10% volume fraction as a function of shear rate for several electric field strengths, showing typical ER behavior. The ER behavior was observed through a controlled shear rate test with electric field strengths of 0–2.0 kV/mm and a shear rate range of 0.01–200 s^{-1} on a log-log plot. As shown in Fig. 5(a), the nano-sized silica-based ER fluid exhibited Newtonian fluid behavior in the absence of an electric field, while it behaved as-like a Bingham model fluid under external electric fields possessing a yield stress. The yield stress is thought to be originated from formed particle clusters and chains by an attractive polarization force. In addition, the shear stresses increased with increasing elec-

tric field strength and became stable over the entire shear stress range when the electric field reached 2.0 kV/mm. Solid lines following the trend of the shear stress curves were generated from a flow curve equation known as the Cho-Choi-Jhon (CCJ) model [33]. As shown in Eq. (1), the CCJ model was introduced to analyze the shear stress trend distinctly using six parameters:

$$\tau = \frac{\tau_y}{1 + (t_1 \dot{\gamma})^\alpha} + \eta_\infty \left(1 + \frac{1}{(t_2 \dot{\gamma})^\beta} \right) \dot{\gamma} \quad (1)$$

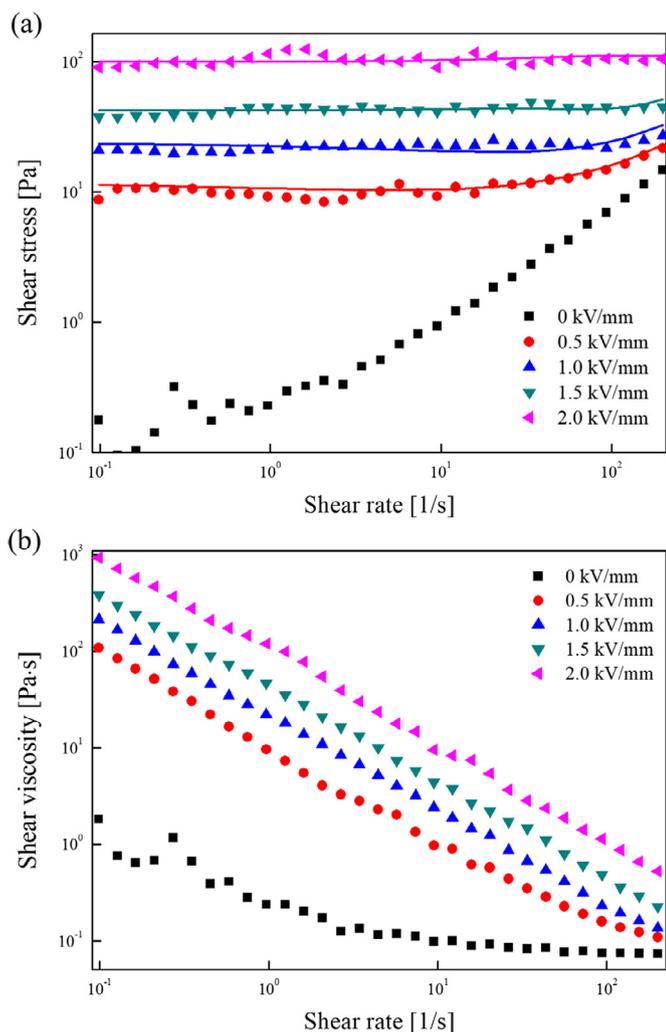


Fig. 5. Flow curve of nano-sized silica (10 vol%) ER fluid under various electric field strengths: Shear stress (a), and shear viscosity (b) curves as a function of shear rate.

Table 1

CCJ model parameters in Eq. (1) for nano-sized silica based ER fluid.

Parameter	τ_y	t_1	α	η_∞	t_2	β
0.5 kV/mm	8.7	0.8	0.25	0.1085	0.01	0.97
1.0 kV/mm	20.9	0.008	0.5	0.1355	0.03	0.97
1.5 kV/mm	39.2	0.0079	1.5	0.225	0.08	0.98
2.0 kV/mm	97	0.005	2.0	0.5247	0.1	0.99

where τ_y is the dynamic yield stress and η_∞ is shear viscosity at an infinite shear rate. Among the six parameters, these two parameters are measurable; the other parameters can be obtained from Eq. (1). t_1 and t_2 are time values, and the α is associated with the decrease at the low shear rate region, whereas the β value is in the range of 0–1 as $d\tau/d\dot{\gamma} \geq 0$. The optimal parameters are summarized in Table 1.

Fig. 5(b) presents that in absence of an electric field, the ER fluid showed a slight deviation from a Newtonian fluid behavior at a low shear rate region, but it became a Newtonian fluid with a constant shear viscosity at a high shear rate region. The fluid also showed typical shear thinning behavior, where the shear viscosity decreased with increasing shear rate under various electric field strengths. This suggests that the initial chain structure formed by the particles was deformed and broken by the shear flow, but still maintained a layered structure on a short length scale.

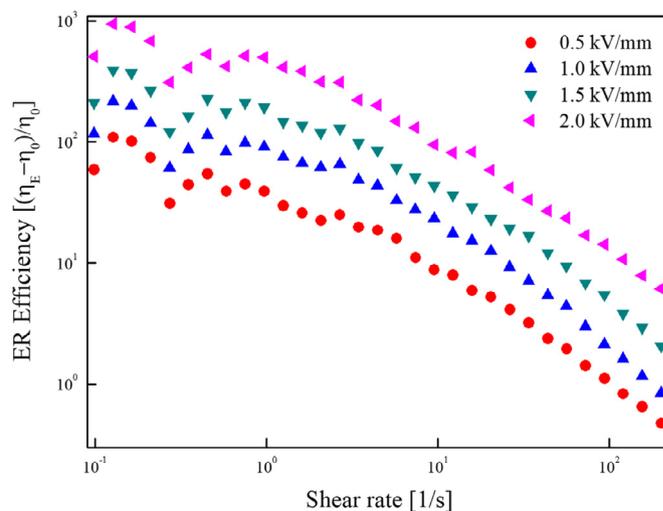


Fig. 6. ER efficiency as function of shear rate of nano-sized silica (10 vol%) ER fluid under various electric field strengths.

The ER efficiency is a significant factor in the ER systems, evaluating the change in the system both in the absence and presence of an electric field. This can be calculated as the following Eq. (2) [34]:

$$\text{ER efficiency} = \frac{(\eta_E - \eta_0)}{\eta_0} \quad (2)$$

Where the η_E and η_0 are the shear viscosities in the presence of an electric field and the field-off viscosity, respectively. Fig. 6 illustrates the ER efficiency of nano-sized silica-based ER fluid under various electric field. As seen, the ER efficiency decreased with increasing a shear rate. This can be attributed to the fact that the particle chains were gradually destroyed, and as a result their resistance to the flow decreased. On the other hand, with increasing electric field strength, the ER efficiency increased.

Strain amplitude sweep tests were carried out initially to determine a linear viscoelastic region (γ_{LVE}) at a constant frequency value of 6.28 rad/s in a strain range of 0.001–100%. Fig. 7(a) shows the storage modulus (G') as a function of a shear strain with an electric field. At the low strain region, the storage modulus was independent of the strain within the LVE region, and a certain strain (0.002%) was chosen for the angular frequency sweep test. When an electric field was applied, the ER fluid exhibited a phase-transition from liquid-like to solid-like, with fibril-like structures formed during this transition.

Furthermore, the progressive structural breakdown could be explained by examining the change in magnitude of the elastic stress ($\tau' = G'\gamma$) as a function of the strain amplitude. At a low strain amplitude ($\gamma < 0.1\%$), the elastic stress increased linearly with increasing strain within the LVE region (Fig. 7(b)), reaching a maximum value (critical strain: γ_c), which suggests the structural breakdown and yield of the ER fluid.

Fig. 8 shows both the dynamic and elastic yield stresses (τ_y) under various electric fields. First, the dynamic yield stress was obtained from controlled shear rate (CSR) tests from the shear stress extrapolation in an extremely low shear rate limit (Fig. 5(a)). On the other hand, the elastic yield stress was deduced from dynamic oscillation measurements with a strain amplitude sweep test (Fig. 7(b)). A simple power-law equation shows the relation between yield stress and an electric field strength as

$$\tau_y \propto E^\alpha \quad (3)$$

where the exponent α can be obtained from an experimental logarithmic fit, and its theoretical value is 1.5 based on the

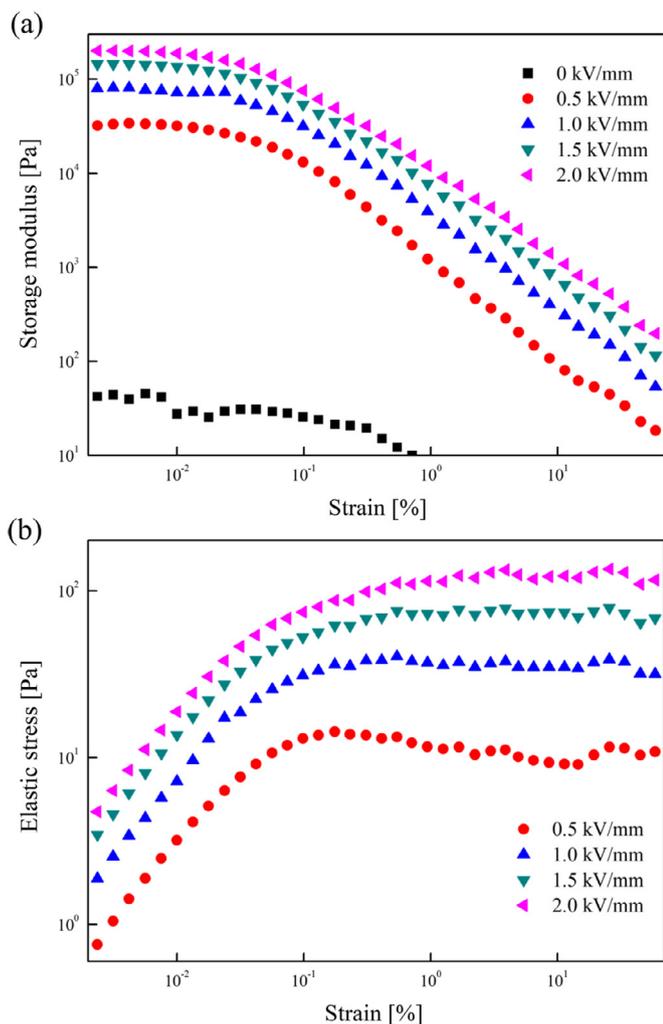


Fig. 7. Storage modulus (G') (a) and elastic stress ($\tau' = G'\gamma$) (b) as a function of strain of the nano-sized silica-based ER fluid.

conduction model, and 2.0 for the polarization model. The slope of both lines of the nano-sized silica particle-based ER fluid was observed to be 1.5, indicating a mechanism following the conduction model [35]. Consequently, for the ER fluid in this study, the dynamic yield stress from the controlled shear rate test was slightly higher than the elastic yield stress from the strain amplitude sweep test.

Fig. 9 presents the frequency sweep data of the nano-sized silica-based ER fluid at a fixed strain of 0.002% from γ_{LVE} , over a range of 1–200 rad/s under various electric fields. Without an electric field, the values of G' and G'' were very similar, indicating its gel-like state. Both G' and G'' increased with increasing electric field strength. The storage moduli were higher than the loss moduli, showing that the solid-like behavior is dominant over viscous behavior. Furthermore, the constant values of the storage modulus showed that the fibril structure of the nano-sized silica-based ER fluid was not broken over this range of angular frequencies. Therefore, the ER fluid behaved as a viscoelastic solid-like material with vibration damping capability by exhibiting the dominant elastic property over the viscous property.

The solid-like properties of the nano-sized silica-based ER fluid can also be analyzed using the data from angular frequency sweep tests. Fig. 10 shows a relaxation modulus as a function of time. The solid-like properties of this ER fluid arise from the storage and loss

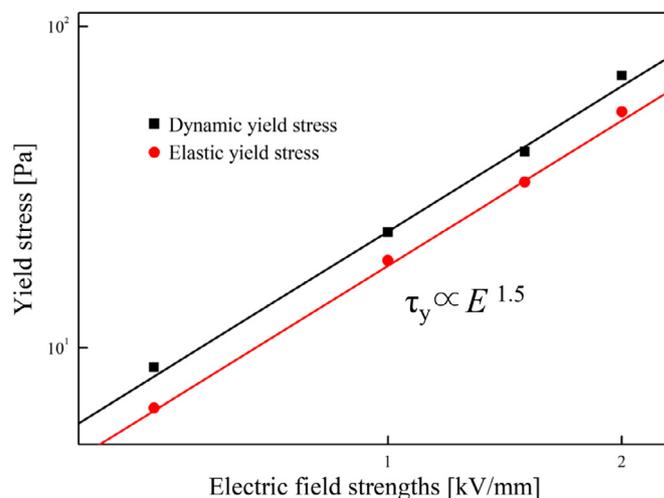


Fig. 8. Dynamic and elastic yield stress as a function of electric field strengths for the nano-sized silica-based ER fluid (10 vol%) under various electric field strengths.

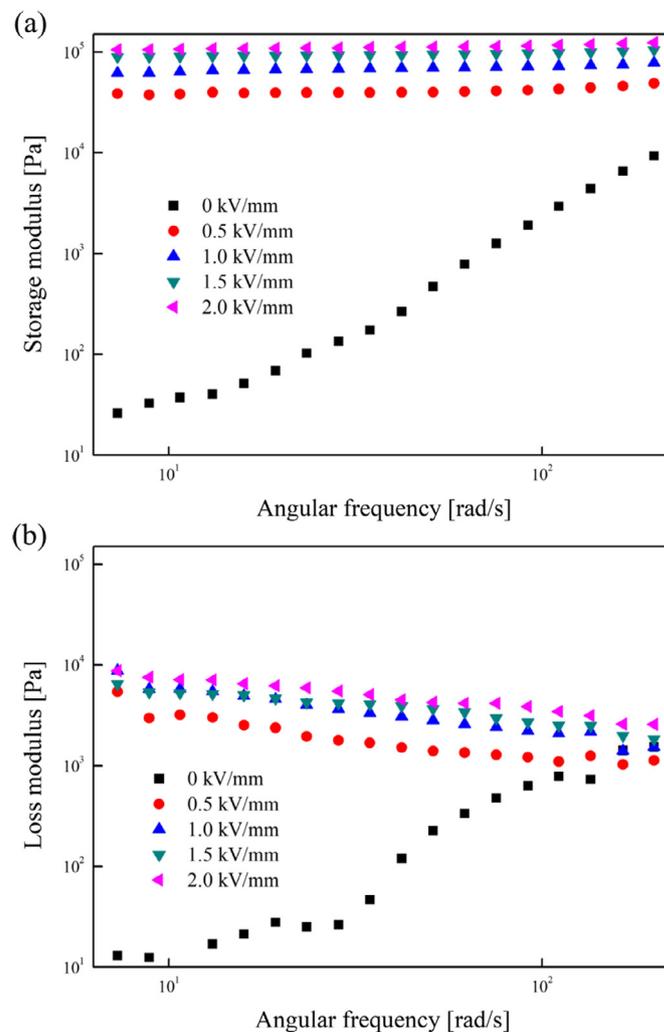


Fig. 9. Storage (a) and loss (b) modulus as a function of angular frequency for the nano-sized silica (10 vol%) ER fluid with a fixed strain, 0.002% under various electric field strengths.

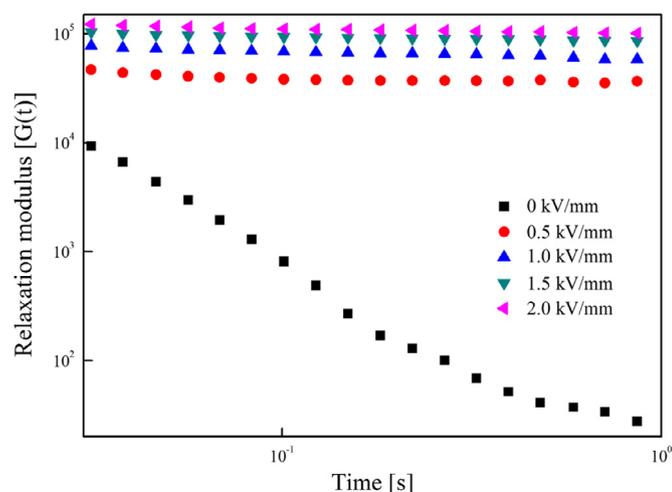


Fig. 10. Relaxation modulus of the nano-sized silica-based ER fluid calculated from storage and loss modulus.

modulus using the following equation:

$$G(t) \cong G'(\omega) - 0.560G''(\omega/2) + 0.200G''(\omega) \quad (4)$$

This is known as the Schwarzl equation [36]. $G(t)$ represents the time-dependent relaxation modulus behavior of the nano-sized silica-based ER fluid. $G(t)$ increased with increasing electric field strength, indicating that the nano-sized silica-based ER fluid exhibits solid-like behavior under different electric fields.

4. Conclusion

Nano silica particles were extracted from rice husk via a facile thermal method and used as a dispersion phase in an ER fluid. The rheological properties of the nano silica-based ER fluid when suspended in silicone oil were investigated using a rotation rheological testing machine under different electric field strengths from 0 to 2 kV/mm. The nano silica particles exhibited excellent ER performance and can be considered a promising component in ER fluids.

Conflicts of interest

The authors declare no conflict of interest.

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