

Review Article

Graphene and Graphene Oxide Composites and Their Electrorheological Applications

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Graphene oxide (GO) based composite systems have been fabricated and investigated as a novel electroresponsive electrorheological (ER) dispersed phase because of their proper electrical conductivity and polarizability for their ER application, in addition to graphene composites. This paper briefly reviews mechanisms of the fabrication of various graphene and GO based composites and their critical ER characteristics including flow curve, yield stress, and dynamic properties measured using a rotational rheometer. Relaxation time and achievable polarizability from dielectric analysis using a LCR meter are also discussed.

1. Introduction

It is valuable to control the physical or chemical properties of materials via various external stimuli such as temperature, light, pH, strain, stress, and electric or magnetic fields, mainly because of their wide range of potential applications [1–5]. Among these, electroresponsive electrorheological (ER) fluid, generally composed of polarizable particles dispersed in an insulating medium, is a kind of intelligent materials whose rheological properties can be rapidly and reversibly transmitted upon the exposure to an external electric field, resulting from the formation of column-like structure of the dispersed particles [6–10]. The unique electroresponsive characteristics endow ER fluids with potential applications in various mechanical devices, such as engine mounts, dampers, clutches, and valves [11–13]. Until now, researchers have developed a range of particulates for ER dispersed phase, such as inorganic hybrids, carbonaceous materials, semiconductive organics and polymers, and mesoporous particles [14–18]. The conductive polymers such as polyaniline (PANI), poly(*p*-phenylene) (PPP), polypyrrole, and polythiophene possessing appropriate electrical conductivity and physical or chemical properties were studied as ER materials [19–22]. Various inorganic or polymer composites with well-designed morphology with high dielectric constant have been also

reported as ER dispersed phases, including anhydrous TiO₂ derivatives, silica coated carbon nanotubes (MWCNTs), and PANI/nanoclay particles [16, 23–27].

Concurrently, graphene, one of the most popular two-dimensional sheetlike nanomaterials, has attracted growing attention in the last ten years as an active component in diverse applications, owing to its high-aspect ratio structure, special layered form, and unique chemical and physical properties. Graphene and graphene oxide (GO) based systems especially have been also employed as ER candidates [28–30]. The elongated anisotropic electroresponsive particles such as rods, sheets, and fibers have been reported to show enhanced ER properties due to their anisotropy, in addition to good dispersion stability. Thereby the graphene nanosheet associated hybrid particles can be considered as potential ER candidates [31, 32] even though its relatively high conductivity is a drawback for its direct application to ER fluids. The high electrical conductivity of the graphene might easily result in an electric short. Therefore, many studies have been focused on fabricating graphene based composite systems as ER candidates. On the other hand, GO sheets can be regarded as an oxidation state of graphene, since they possess huge amount of hydroxyl, carboxylic, and epoxide functional groups on its basal planes and edges. From the XPS survey, the carbon/oxygen ratio of GO sheets was reported to

be about 1.43 [33], and then the C/O atomic ratio increased to 10.3 in the reduced exfoliated graphite oxide which is higher than that of its precursor GO [34]. Thereby it leads to good dispersion stability in aqueous and other organic solvents [35, 36]. In addition, in contrast to pure graphene, GO sheets are more appropriately utilized as ER materials even without any posttreatment because of their relatively reduced electrical conductivity [33, 37]. In addition, their hydrophilic characteristics make them appropriate for fabricating various GO based inorganic or organic composites.

To enhance their ER activity, diverse synthetic mechanisms for fabricating graphene/GO based ER composites, including bare GO sheets, graphene nanosheets/layered double hydroxide (LDH) composites, graphene/chitosan nanocomposites, graphene/carbonaceous composites, GO-wrapped mesoporous silica spheres, silica anchored GO composites, GO/PANI nanocomposite, GO/Al₂O₃ particles, GO/TiO₂ nanocomposites, and core-shell structured polystyrene (PS)/GO particles [38–44], have been introduced. Note that while the pristine GO itself still lacks sufficient ER performance due to not only slightly high electrical conductivity compared to conventional ER materials but also its flexible sheet structure, its hybrid with various inorganics and polymers demonstrates enhanced ER performance compared to their pristine states.

In this short review paper, we mainly focus on various mechanisms for the preparation of graphene and GO based ER dispersed phases. Typical analysis of their ER properties including flow curve, dynamic yield stress, viscoelastic properties, and dielectric constant is also discussed.

2. Materials and Fabrication Mechanism

2.1. Solvent Exchange Method. As above mentioned, pure GO sheet could be a favorable candidate for the preparation of ER dispersed phase because of its reduced electrical conductivity and high polarizable properties resulting from the presence of functional groups on its edges and planes. However, the restacking properties of GO sheets might cause their poor dispersion stability in silicone oil after high shearing process and correspondingly restrain their further applications in engineering fields.

To enhance the dispersion stability of GO sheets in nonpolar liquids, Hong and Jang [33] introduced a solvent exchange strategy for dispersing GO sheets in silicone oil stably. As shown in Figure 1, the isolated GO sheets aggregate and restack together after a drying process, resulting in poor stability for being redispersed in silicone oil. The GO sheets were initially fabricated using a modified Hummers method. On the other hand, as for type II, the suspension of GO sheets dispersing in ethanol was added to the silicone oil, and then the mixture was separated using a centrifugation process. The GO sheets can be thus settled in the silicone oil, obtaining highly stable GO suspension in silicone oil. No serious sedimentation of the solvent exchanged GO suspension was observed even after 3 months [28].

2.2. Mechanochemical Method. Shin et al. [28] have proposed a highly dispersible GO colloidal suspension prepared

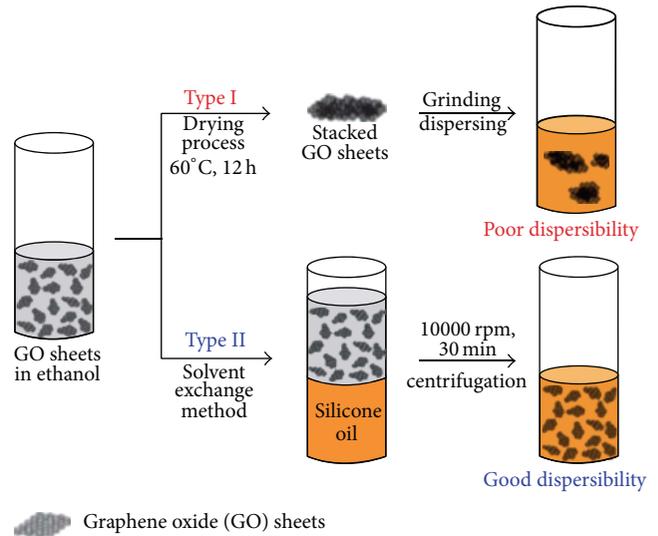


FIGURE 1: Illustration of different procedures for fabricating GO based ER fluids [33].

from pristine graphite flake using a mechanochemical (ball-milled) process. The pristine graphite was placed in a ball-milled capsule loaded with different sizes of metal balls, and then the capsule was fixed and agitated at 550 rpm for 2 or 3 hours. The resulting ball-milled graphite powder was collected using a sieve to separate the metal balls. The size of the obtained graphene sheets can be controllable by changing the ball-milling time. In addition, the obtained graphene sheets were oxidized to GO using a modified Hummers method, and the presence of the functional groups on the GO sheets generates electrostatic repulsion, which limits the restacking of GO sheets and improves the suspension stability of the GO based colloids accordingly. The obtained GO sheet based ER fluids show high ER activity and fast responsive time under external electric field compared to those of the GO sheets without the ball-milling process.

2.3. In Situ Polymerization. The typical conducting polymers such as PANI and polypyrrole and their composite systems have been extensively studied, thanks to their controllable electric conductivity and other attractive advantages such as bulk processability and dielectric characteristics [45–47]. PANI particles or PANI based hybrids with different morphologies have been extensively studied because of their low cost, facile fabrication, and attractive environment stability. Owing to these properties, PANI based composites have been employed as a dispersed phase of electroresponsive ER materials. However, the high electrical conductivity of PANI causes a problem of electric short during the measurements and obstructs its further engineering applications as for ER materials. In order to overcome these problems, PANI based hybrids have been fabricated, such as PANI granule coated graphene composites, PANI/TiO₂ composites, PANI/vanadium oxide particles, and PANI nanofiber/kaolinite nanoparticles [26, 48–51].

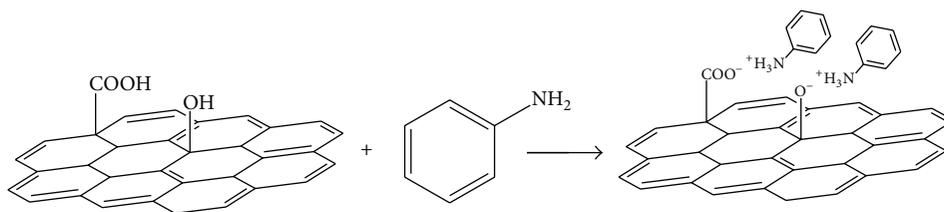


FIGURE 2: Scheme of the formation of GO/PANI composites via electrostatic attractions [52].

As shown in Figure 2, the PANI granules were synthesized in the presence of GO suspensions using ammonium persulfate as an initiator, thus resulting in GO/PANI composites obtained by an in situ polymerization method [52].

2.4. Electrostatic Attraction. Naturally, the GO sheets possess negative electric charges due to the presence of functional groups, and, therefore, a facile electrostatic interaction associated approach can be employed to prepare diverse GO based composites [15, 40, 53]. Goswami et al. [51] synthesized the GO-wrapped titania microspheres through an electrostatic attraction method. The presence of GO sheets introduced enhanced interfacial polarization to the ER fluid compared with that of pure titania microspheres. Lee et al. [15] have coated the polar GO sheets onto the surface of modified silica rods via electrostatic attraction. Silica particles were firstly treated with 3-aminopropyltrimethoxysilane for 12 h to introduce amine groups, resulting in their positively charged surface [15], and then the GO sheets were added to the suspension of silica particles to afford GO-sheet-wrapped silica particles. The effect of geometry of the particles on ER characteristics was also studied since it is well known that the aspect ratio plays a positive role in improving the ER effects. For three different particles of the GO-wrapped silica sphere, the rod with an aspect ratio of 5, the rod with an aspect ratio of 20, and the ER materials with a high-aspect ratio were observed to exhibit superior ER performance [15], based on the fact that three factors of flow resistance, mechanical stability, and dielectric properties of the ER fluids increased as the aspect ratio of the ER materials increased, thus demonstrating higher ER efficiency.

2.5. Microwave-Assisted Method. Microwave-assisted method for materials' fabrication is in general a time-saving and facile strategy to prepare diverse materials with different morphology and phase [54, 55]. Hu et al. [39] reported a simple strategy for preparing chitosan-decorated graphene nanosheets prepared using a microwave irradiation method. The graphene layers were coated by chitosan particles and GO sheets were reduced to graphene simultaneously. The chitosan-decorated graphene nanocomposites exhibited a noticeable ER behavior under an applied electric field strength. Novel water-free dry-based ER systems consisting of poly(ionic liquid) particles were also fabricated using a microwave-assisted polymerization strategy [56].

2.6. Pickering Emulsion Polymerization. Pickering emulsion is a well-known process of eco-friendly emulsion systems

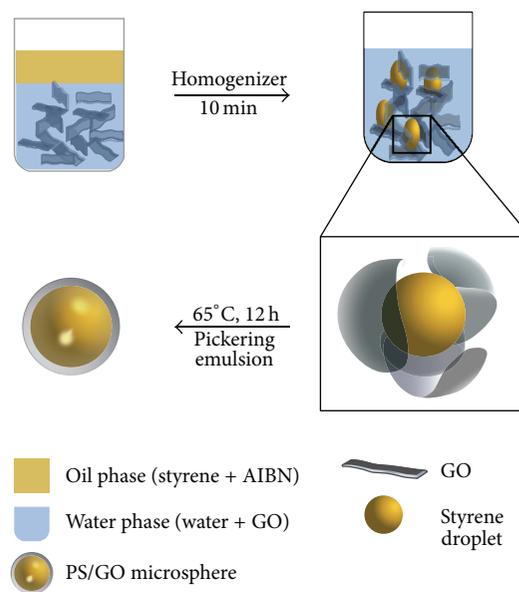


FIGURE 3: Scheme of the preparation of PS/GO microspheres using the Pickering emulsion polymerization [41].

stabilized by solid particles instead of conventional organic surfactants [57, 58]. Various inorganic particles, such as silica, laponite or LDH clays, TiO_2 , ZnO_2 , and magnetic Fe_2O_3 and Fe_3O_4 nanoparticles, are frequently employed as solid stabilizers in the Pickering emulsion polymerization [59–62].

Recently, GO sheets were used as solid stabilizer in Pickering emulsions because of their intrinsic amphiphilic properties [63–65]. Kim et al. [41] developed core-shell structured polystyrene (PS)/GO microspheres using a Pickering emulsion polymerization and investigated their ER characteristics using a rotational rheometer as shown in Figure 3. Introduction of the PS microspheres controls the electrical conductivity of the obtained composites properly for the ER measurements. On the other hand, when magnetic Fe_2O_3 nanoparticles were applied as a stabilizer for Pickering emulsions with the polymerization of PS, the obtained PS/ Fe_2O_3 hybrid particles are reported to exhibit magneto-responsive magnetorheological (MR) characteristics under an applied magnetic field strength [66]. Note that magnetically analogous MR fluids behave similarly with ER fluids under external magnetic fields [67].

2.7. Sol-Gel Method. A sol-gel method is another facile strategy to prepare diverse inorganic particles or inorganic

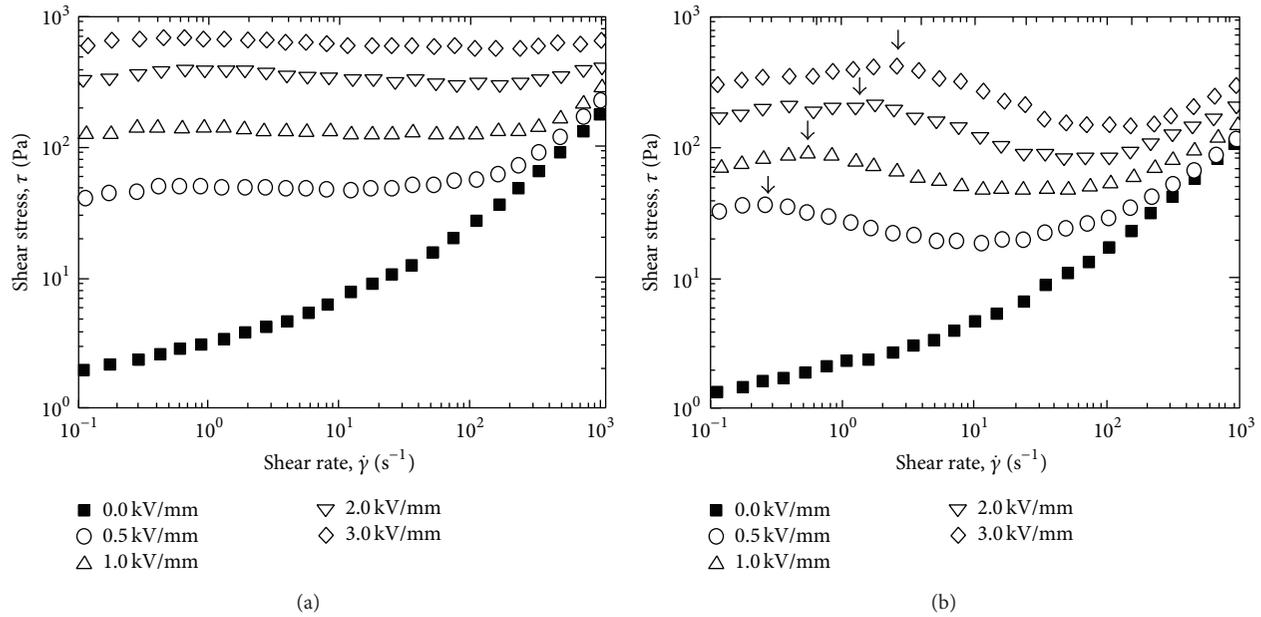


FIGURE 4: Shear stress versus shear rate of (a) GO-wrapped titania microspheres and (b) bare titania microspheres based ER suspensions [53].

material based composites [16, 29, 68–71]. To enhance the ER effect, a growing number of graphene based components with high dielectric constant substances have been reported. Yin et al. [44] coated mesoporous silica particles on the graphene nanosheets using a sol-gel method by the hydrolysis of tetraethylorthosilicate in the presence of GO colloids. The introduction of silica particles could solve the high electric conductivity issue of pure graphene and enhance the stability of the resultant composites in silicone oil. Furthermore, Hong et al. [29] modified graphene sheets using TiO_2 nanorods by a facile nonhydrolytic sol-gel reaction, with which the higher dielectric properties and fast responsive reaction were observed.

Through this review paper, it is found that diverse mechanisms and methods have been employed to synthesize electroresponsive graphene or GO based potential ER particles. Compared to the simple physical method, the particles obtained using chemical approach can be more stable in general. The in situ polymerization is considered to be a facile and fast way to prepare various composites; nonetheless, it is difficult to design the particles with well-controlled morphology. It is available to acquire composite particles using a positive-negative attraction, and the surface modified procedure requires organic surfactants which could bring a negative effect to the ER systems. Microwave-assisted process is a novel and time-saving process; however, the vigorous microwave irradiation may destroy the structure of the materials. Recently introduced eco-friendly Pickering emulsion polymerization has attracted increasing attention for ER and MR community owing to its free-organic surfactant mechanism, while the selection of suitable solid stabilizer particle is also a challenge. Sol-gel method is convenient to synthesize various particles with different particle size; unluckily, not all of the particles can be obtained using the sol-gel method.

3. ER Characteristics

The typical ER behaviors of the graphene or GO based ER systems including flow curves (shear stress and shear viscosity versus shear rate in a controlled shear rate test mode), on-off switch at a fixed shear rate, dynamic test, and the dielectric constant parameters such as achievable dielectric constant and relaxation time have been extensively investigated.

Figure 4 shows the comparison of flow curves of shear stress versus shear rate for GO-wrapped TiO_2 and pure TiO_2 microsphere based ER fluids [53]. At a free electric field, the shear stress increases almost linearly as a function of shear rate, confirming the Newtonian properties of these two kinds of ER fluids. However, when exposed to an external electric field, the shear stress increases abruptly exhibiting solid-like properties, at the same time generating a yield stress. As expected, the GO coated TiO_2 microsphere shows higher shear stress at the same electric field strength, proving the positive effect of GO sheets on improving ER activities.

Figure 5 shows shear stress of both graphene nanosheet/LDH and pure LDH based ER suspensions with and without the stimuli of AC electric field [38]. The shear stress of the suspensions increases rapidly under the stimuli of external electric field and rapidly drops back to the original values when the electric field was removed. In addition, the shear stress increases when the electric field strength increases, and the values of shear stress for the graphene nanosheet/LDH suspension are almost 2.5 times higher than those of the pure LDH suspension at the same external electric field strength. The results verify that the graphene nanosheet/LDH composite suspension possesses a higher ER effect compared to that of the pure LDH sheets based ER suspension.

The relationship between dynamic yield stress (τ_y) and electric field strength (E) can be investigated to evaluate

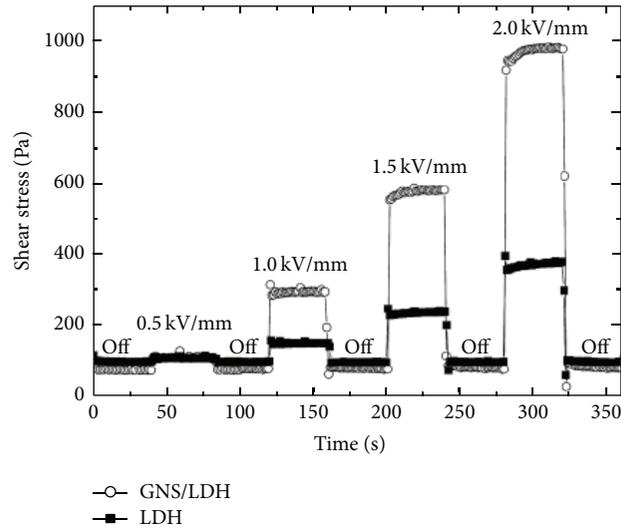


FIGURE 5: The comparison of the shear stress for graphene nanosheets/LDH and pure LDH based suspensions [38].

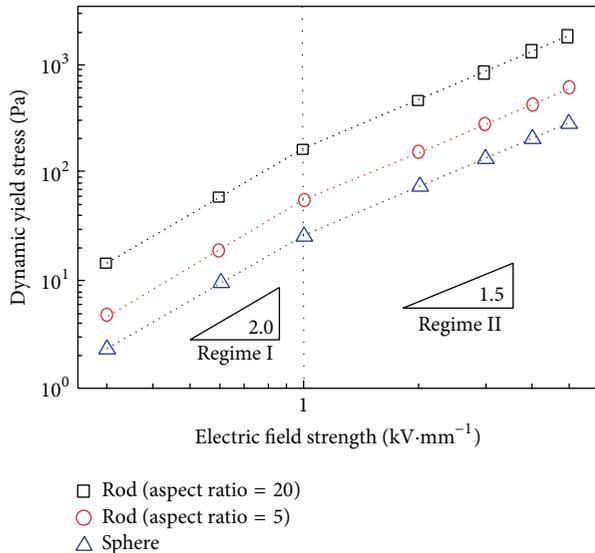


FIGURE 6: Dynamic yield stress versus electric field strength of diverse GO-wrapped silica components based ER fluids [15].

the ER properties more accurately [72, 73]. As shown in Figure 6, the dynamic yield stress increases linearly with the electric field strength. When the applied electric field was less than 1 kV/mm, the slope is 2.0, indicating the polarization model. The slope declines to 1.5 when the electric field is higher than 1 kV/mm, which is called conduction model [74, 75]. In particular, the ER fluid with enhanced aspect ratio exhibited high dynamic yield stress.

The dielectric property of ER materials is also a critical factor for ER activities. It is widely accepted that good ER materials should have a large achievable polarizability and short relaxation time. Figure 7 presents the dielectric spectra (ϵ' and ϵ'') for TiO₂/graphene and GO particles based ER fluids as a function of frequency. The Cole-Cole

equation was introduced to fit the dielectric properties of the TiO₂/graphene and GO based ER fluids [29, 42]:

$$\epsilon^* = \epsilon' + i\epsilon'' = \epsilon_\infty + \frac{\Delta\epsilon}{(1 + i\omega\lambda)^{1-\alpha}} \quad (0 \leq \alpha < 1). \quad (1)$$

Here, ϵ' is the dielectric permittivity, ϵ'' is the dielectric loss factor, ω is the angular frequency, and λ is the relaxation time [76, 77]. The large achievable polarizability and fast relaxation time of the TiO₂/graphene composites based ER fluid give positive effect on ER activities, confirming the good ER behavior of TiO₂ particles.

As mentioned, the GO sheets could restack together, resulting in poor dispersibility in silicone oil; therefore, it is desirable to improve the stability of GO suspensions. Shin et al. [28] have studied the sedimentation ratio of the ball-milled GO sheets in silicone oil compared with bare GO sheets without ball-milling process. As shown in Figure 8, the GO suspensions after ball-milling exhibited enhanced stability with increasing ball-milling time. The GO suspension with 3 h ball-milling process was found to be stable even after 90 days.

4. Conclusions

This short review paper summarizes mainly various mechanisms for preparing graphene and GO based ER particles, including solvent exchange method, mechanochemical method, in situ polymerization, electrostatic attraction, microwave-assisted method, Pickering emulsion polymerization, and sol-gel method. The combination of diverse inorganic or organic materials can be well designed with different morphologies and potential applications using these methods. Their typical ER properties measured from a rotational rheometer, dielectric constant, and physical or chemical properties are also discussed for their own distinctive ER performance. The GO based composites processing

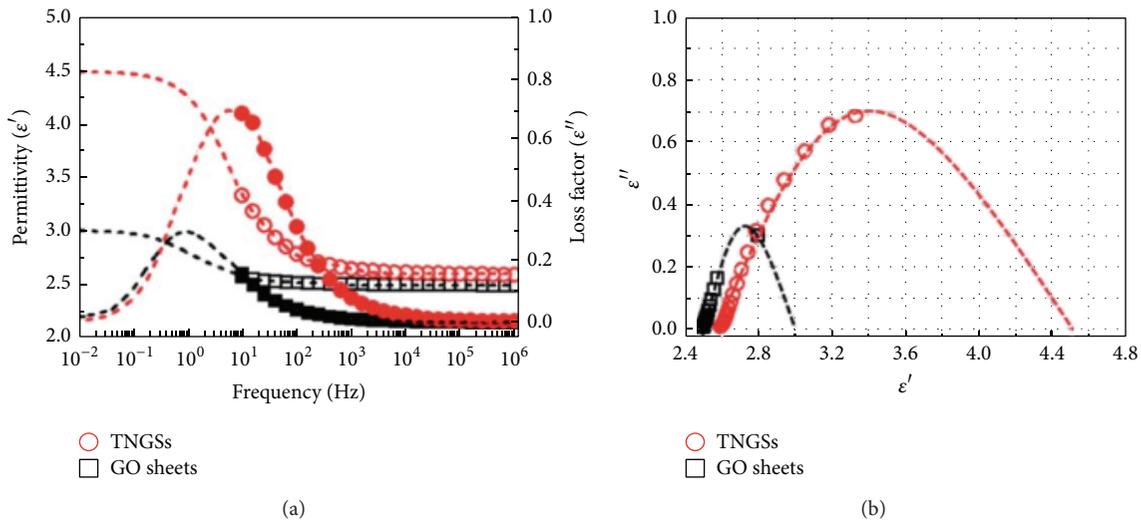


FIGURE 7: (a) Dielectric constant and (b) Cole-Cole plots for TiO_2 /graphene and GO based ER fluids [29].

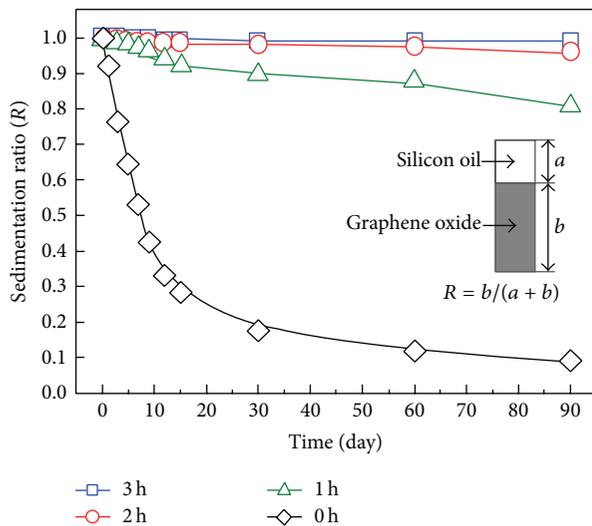


FIGURE 8: Sedimentation tests of GO based ER fluids with different ball-milled time [28].

high polarization and good stabilization will trigger more and more attractions in different areas in the future.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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