

Rheology of Fumed Silica and Polyethylene Glycol Shear Thickening Suspension with Nano-clay as an Additive

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ABSTRACT

Shear thickening properties of fumed silica-polyethylene glycol (PEG) with shear thickening fluid (STF) of different concentrations and with an organically modified clay, Nanomer I.28 E as nano-additive have been investigated by both steady-state and dynamic state rheology. Difference in rheology if instead of nanoclay, an equal wt% of additional fumed silica is added to 20 per cent fumed silica-PEG200 STF, has been studied. At 25 °C, in case of addition of nanoclay the increase in critical viscosity is less than that observed for same additional amount of fumed silica. Interestingly, an opposite result is seen at higher temperatures i.e. 45 °C and 55 °C. Moreover, the difference in steady-state and dynamic state viscosity values decreases on addition of nanoclay. It is noted that an increase in concentration of clay increases the value of dynamic parameters whereas for STF of only fumed silica particles the values are constant irrespective of the change in concentration. More importantly, ease of processing, elasticity, stability and consistency of rheological results of STF increases to a significant extent on addition of relatively inexpensive nano-additive.

Keywords: Shear thickening fluids; Rheology; Viscoelasticity

1. INTRODUCTION

Shear thickening fluids (STF) constitutes a subgroup of smart fluids, which shows a dilatant, non-Newtonian fluid behaviour. When subjected to an impact, say of a high velocity projectile, these materials exhibit a reversible and discontinuous increase in viscosity¹⁻³. STF are composed of dispersions of rigid particles of nano or micron range dimensions in a carrier fluid which is mostly a polymer⁴⁻⁶. Energy absorption, energy dissipation and energy channeling capabilities of these fluids make them suitable for various protective applications in industry, biomedical, and military etc⁶⁻⁸. There has been a lot of research on high performance aramid fabrics such as Kevlar impregnated with STF⁹⁻¹¹. Such a system as body armour can provide rigidity and stiffness to resist high speed projectiles like bullets, knife blows, and other similar attacks yet allowing the flexibility and reduced weight to the wearer. Similarly, research is also going on for STF integration with extra-vehicular activity (EVA) suit for protection against micrometeoroids and orbital debris (MMOD)¹².

The steady-state rheological characteristics of STF in terms of important parameters like critical viscosity (maximum viscosity) and critical shear rate (shear rate at the transition) and the dynamic state properties under oscillatory shear in terms of loss modulus, storage modulus, and complex viscosity

as a function of frequency and strain have been reported in literature^{13,14}. Among the parameters affecting the shear-thickening behaviour, shape of the particles has been one of the major contributing parameter with rod and plate like particles showing greater critical viscosity than grains and spheres¹⁵⁻¹⁹. Petel²⁰, *et al.* suggested that STF consisting of only spherical silica is not optimum for ballistic applications given its low mechanical and thermal properties. Jogun & Zukoski²¹ have shown that for clay particles having large aspect-ratio, the excluded volume is much larger than its actual volume and more than that of micron-sized spherical particles⁵. As a result, for the two phase dispersion system of clay as nano-additive in fumed silica-PEG STF the particle motion in the STF will be restricted at much lower volume fraction than the maximum packing fraction of the particles. This is important as it lowers both the cost and weight of the STF. Additionally, the addition of clay particles can increase dispersibility of nanoparticles in the suspension by enhancing the interaction among the particles and resulting in a better clustering of particles. Moreover, fumed silica nano-particles (11 nm - 14 nm) can exhibit very high intensity shear thickening compared to those of simple silica (100 nm - 600 nm) suspensions and that too at much lower loadings (minimum 18 per cent weight fraction)^{8,22,23}. The motivation in the present study is to study the rheology of suspensions containing fractal fumed silica particles and high aspect ratio nano-scale clay as additive.

2. EXPERIMENTAL

2.1 Materials

All the constituents were purchased from Sigma Aldrich, India. Fumed nanosilica S5380 (size 11 nm; purity $\geq 99\%$; specific surface area 255 m²/g) was used as the dispersed phase; the suspending carrier material was polyethylene glycol 200. Nanomer I.28 E, a bentonite clay with its surface modified with quaternary trimethyl stearyl ammonium with cation exchange capacity of 93.7 meq / 100 g was used as a nano-additive in STF.

2.2 STF Preparation

Nano-dispersion in a matrix is a critical step, which was achieved by the probe sonication technique as shown in Fig. S1(a) and S1(b) of supplementary data. A sonication time of 10 min by a probe sonicator (ultrasonication probe of 6 mm dia, power 750 W and frequency 20 KHz) at 40 per cent amplitude was found to be optimum for fumed silica-PEG system of 20ml volume. For samples containing nanomer clay as nanofiller, the suspension of clay and PEG were first sonicated for 2 min prior to addition of nanosilica to break the structure comprising of stacked platelets otherwise coagulation can occur (Fig. S1(c)). The concentration of clay particles less than or equal to 5 per cent by weight concentration facilitates proper exfoliation/intercalation of high aspect ratio clay particles²⁴. Therefore, clay particles were added at low concentrations of 3, 4 and 5 phr (parts per hundred of resin) in 20 wt% STF of fumed silica and PEG (Fig. S2) corresponding to 2.9, 3.85, and 4.76 weight % of clay in base STF.

2.3 Rheological Measurements

Rheological analysis was done by using Rheometer (MCR52, ANTON PAAR) with a cone and plate geometry, with a plate diameter of 40 mm and a cone angle of 2°. All steady-state rheological measurements were conducted at wide range of shear rates (1 s⁻¹ to 2000 s⁻¹) and temperatures (25 °C to 55 °C). An excellent temperature control is provided by the Peltier element inbuilt in the rheometer. The dynamic rheological tests were done for strains from 10 per cent to 1000 per cent at 25° to determine the storage, elastic or recoverable response and dissipative, viscous or damping response of the STF. Once the high speed projectile hits the STF, the initial response is by the STF in fluid state and very quickly it transforms to a semi-solid to solid-like state. The former response has been studied through steady-state rheology giving critical shear rates and the latter by dynamic experiments which would give critical strain.

For typical STF applications, the time-scale of deformation is also very important. Therefore, study of dynamic rheology at different frequencies was also studied. Preshearing alleviates the problem of poor reproducibility and therefore a preshearing protocol of 0.1 to 1s⁻¹ for steady-state and 1 per cent to 10 per cent strain for dynamic-state rheology respectively for 60 s was followed for every rheological measurements.

3. RESULTS AND DISCUSSIONS

3.1 Characterisation

The particle dispersion was determined through

Transmission Electron Microscopy (Model- FEI Tecnai G² 20 S-Twin). For TEM analysis, samples were sonicated with ethanol which was twice the amount of that of the sample, for 30 min and then a small drop of solution was placed onto a Cu grid and allowed to evaporate, typically under vacuum before being viewed. Figure 1 depicts the TEM image of prepared STF sample of 20 per cent fumed silica. The fractal fumed silica particles were nearly spherical in shape and the size distribution was 13-15 nm.

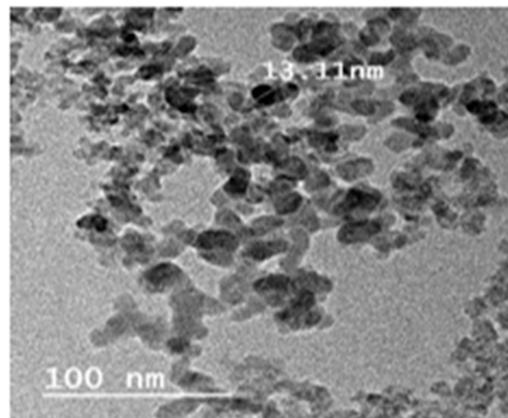


Figure 1. TEM image of 20 per cent STF.

3.2 Stability

For reproducible results and performance, the STF suspension should be stable for a reasonable long time period i.e dispersed particles should not agglomerate. However, clay is known to have a high settling tendency in suspensions. Interestingly, all compositions of STF in the present study were stable for more than a month i.e. no phase separation was seen. It was also noted that at low concentration of 3 phr nanoclay additive, a small amount of deposition of clay can be seen visually after a month or more that leads to decrease in shear thickening but interestingly for high concentration i.e. 4 and 5 phr, no settling was observed. This may be attributed to the possible increased intercalation of PEG molecules in the interlayer spacing of high concentration of clay, compared to low concentration which would stabilise the clay particles and prevent their settling. Additionally, it is interesting to note that when repeating the same experiment thrice, STF containing clay nanofiller gave more reproducible results than fumed-silica STF without clay.

3.3 Steady-state Rheology

Figure 2(a) presents the variation of steady-state viscosity with shear rate for six different mixtures having 22.33, 23, 23.08, 23.81, 26 and 29 wt.% fumed silica in PEG. Figure 2(b) show the viscosity profiles for 3, 4 and 5 phr of nano-clay as additive to 20 wt% fumed silica-PEG STF. It was found that 20 per cent composition (without clay) provided an optimum combination of low viscosity at zero-shear, marked shear thickening, high-post transition viscosity and also ease of dispersion and relative stability with consistency of results. For this reason, 20 per cent concentration of fumed silica-PEG STF was selected as a base STF composition. All the results were obtained at 25 °C.

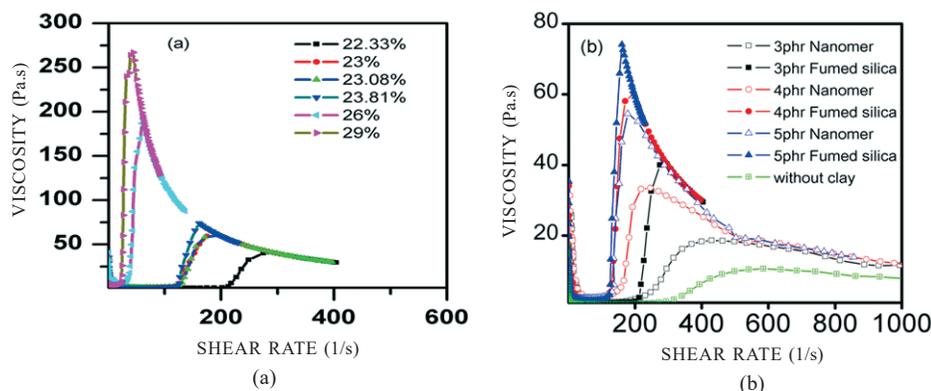


Figure 2. Steady state rheology of (a) different concentration of fumed silica-PEG STF and (b) 20 per cent STF with different add-on % of nano-clay.

It can be seen that three well defined regions are present. The first viscosity region is the shear-thinning region, which is due to arrangement of particles in the direction of flow of the polymeric carrier fluid, followed by a second region where shear-thickening occurs due to the formation of hydroclusters. The third region once again corresponds to shear-thinning region after which no shear-thickening was observed and the nature of viscosity-shear rate plots, particularly the portion after the peak value, are following a typical parabolic nature and are also coinciding for all STF. Thus, the carrier fluid contribution to the viscosity dominates, as confirmed by same decreasing value of viscosity for all the samples having same carrier fluid PEG200. In the second region, as shear rate increases to critical shear rate, viscosity exhibits an abrupt and sharp increase for mono-disperse suspensions (i.e. no clay) as like particles form clusters easily. However, there is a relatively smooth increase for bi-disperse suspensions with the transition spreading over a wide range of shear rates. The possible reason for this is that a greater shear thickening microstructure distortion is exhibited by the fumed silica dispersions as compared with dispersions containing nanoclay as additive. Also, STF with nanoclay have higher critical shear rates when compared with pure fumed silica-PEG STF. Additionally, the rheology confirms the phenomena of Ferris effect that describes that a broader particle size distribution (i.e. clay and nanosilica particles) yields a lower viscosity than a narrow particle size distribution (only fumed silica particles), for the same concentration of particles²⁵.

In organically modified clay the charges between the layers are neutralised that helps to reduce the tendency of clay agglomeration during the dispersion in the suspension medium by reducing interlayer attractive forces and thus aids in intercalation or exfoliation. Moreover, the interaction between the clay platelets and solvent increases. The addition of clay indicates that the network of particles that is formed at the onset of shear thickening is small when compared to monodisperse nano-silica particles but are more interconnected and stronger as suggested by large values of viscosities even at very high shear rates. As seen from Fig. 2(b), the values of critical shear rate are nearly independent of the type of particles used as an additive and depends only on add-on particle concentration

i.e. critical shear rate decreases as concentration increases. However with regard to critical viscosity, the samples with 3 and 5 phr clay showed marked shear thickening and high post-transition viscosity. The probable reason for this behaviour is that critical shear rate changes in a major way with the volume fraction of solids, which is same in both the cases. However, change in the value of critical viscosity is due to the interaction between particles of different shapes in the system. As shown in Table 1, for a given solids concentration, the values of critical viscosity for only fumed silica STF are always higher than those for STF containing clay. This is in agreement with the observations made earlier²⁵.

Table 1. Steady-state rheological parameters

Sample code	Additive	Add on weight (phr)	Critical viscosity (Pa.s)	Critical shear rate (1/s)
3N	Nanomer	3	14	115
3S	Fumed silica	3	40	113
4N	Nanomer	4	33	93
4S	Fumed silica	4	60	87
5N	Nanomer	5	55	54
5S	Fumed silica	5	74	62

3.4 Effect of Temperature

The experiments were also done at 35 °C, 45 °C and 55 °C, to study the effect of temperature on STF behaviour. As temperature increases, the strength of hydrogen bonds between the particles and PEG molecules decreases resulting in a decrease in low shear viscosity. Also, the Brownian forces increases resulting in an increase in repulsive forces, which results in requirement of higher shear rates for formation of hydroclusters. This temperature dependency is consistent with results reported in literature^{26,27}. It is noted that there is a large decrease in values of viscosity for 3 phr of both additives (Fig. 3(a) and 3(c)) as compared to their 5 phr concentration (Fig. 3(b) and 3(d)) at all temperatures. Additionally, it turns out that the 3 phr clay as nano-filler makes the STF retain the high value of viscosity over a large range of shear rates as indicated by a gradual decrease (broad shoulder) in viscosity after the critical viscosity (Fig. 3(c)). Significantly, at high temperatures of 45 °C and 55 °C, the further decrease in critical

viscosity values becomes very significantly negligible for clay as filler as shown in Fig. 3(c) and 3(d). Higher temperatures were thus not probed, and also there is a chance of samples ejected out from the rheometer plate due to very low viscosity in the course of experiment.

3.5 Dynamic State Measurements

The material response (stress /strain) to increasing deformation strain was monitored at a constant frequency and temperature. The strain percent was linearly varied from 1 per cent to 10,000 per cent and the frequencies selected were 10, 30, 50 and 70 rad/s.

The dynamic test results at 25 °C with respect to complex viscosity as shown in Fig. 4. They depict strain thickening behaviour of STF after critical strain amplitude. It can also be seen that this transition occurs at smaller critical strains as the frequency of deformation is increased. However, maximum value of complex viscosity decreases slightly. This agrees well with the reports in literature on STF^{4, 26}. The reason for this may be that at high frequency small strain is sufficient to form hydroclusters subsequently, the less time is available for particles to form large hydro-clusters which results in decrease in maximum value of complex viscosity.

For clay nano-particles as additives, the difference in

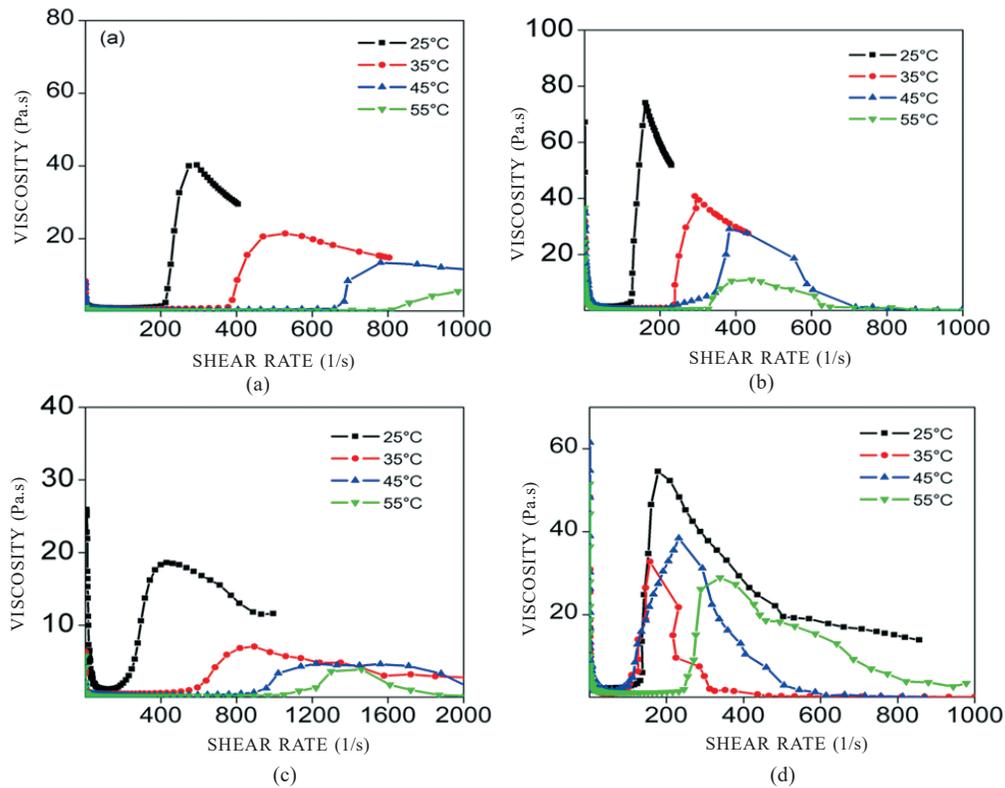


Figure 3. Steady-state rheology of 20 per cent STF as base containing (a) 3 phr fumed silica, (b) 5 phr fumed silica, (c) 3 phr nanomer, and (d) 5 phr nanomer as additive.

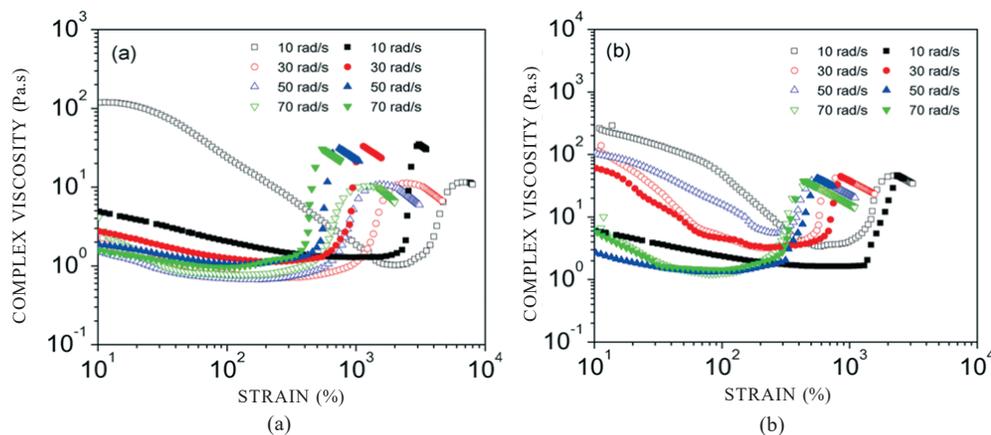


Figure 4. Comparison of complex viscosity and strain at different frequencies (a) 3 phr nanomer (open symbols) and fumed silica (close symbols) and (b) 5 phr nanomer (open symbols) and fumed silica (close symbols).

critical viscosity of steady-state (Fig. 2(b)) and complex viscosity (Fig. 4) at 3 and 5 phr is quite small i.e. around 13 and 55 Pas respectively. On the other hand, for nano-silica at higher concentration of 5 phr, the maximum value of the steady state viscosity (about 75 Pas) is consistently higher than the critical value of complex viscosity (about 60 Pas). The probable reason is that the particle-particle interaction for fumed silica particles would be predominantly higher under steady state conditions than in dynamic state where continuous forming and breaking of particle clusters would prevent viscosity buildup with consistently greater effect for higher loading. Also it can be noted from Fig. 4(b) that the complex viscosity of both 5 phr nanomer and 5 phr fumed silica are almost same. This suggests that in contrast to steady-state deformation, under oscillatory deformation at same high concentration, both fumed silica and nanomer as filler show comparable shear thickening properties. Similar to steady state critical shear rate, the critical strain is also dependent only on the concentration of filler at all frequencies studied except at very low frequency i.e. at 10 rad/s, which suggests that at very low frequency, high strain per cent is required for shear thickening. Also, expectedly the values of storage and loss modulus increases as concentration of fillers increase with a sudden upturn at critical strain. Similar to complex viscosity profile, both moduli show less change with concentration for STF containing only silica as shown in Fig. S3 and Fig. 5. It can be seen from Fig. 5 for 5phr concentration, that in strain sweep at all four frequencies, G' and G'' show three zones similar to that seen in complex viscosity profile i.e. as strain increases initially, G' sharply decreases whereas, G'' slightly decreases and then both moduli increases after critical strain as cluster volume increases. At higher strains, G' values drop as a result of rupture of clusters (Fig. 5(a)), which also suggests the relaxation of the structures of STF. G'' also decreases due to collapsing of clusters and strain thinning starts (Fig. 5(b)). As the constant frequency value increases, the maximum value of G' and G'' increases, which again confirms that on shearing suspension more quickly, it become more elastic and viscous.

It has been noted that the elastic modulus G' , is more sensitive to shear strain than the loss modulus, G'' , and shows highly non-linear response at low shear strains (Fig. 5(a) and 5(b)).

As $G'' > G'$, therefore STF are viscoelastic fluids and

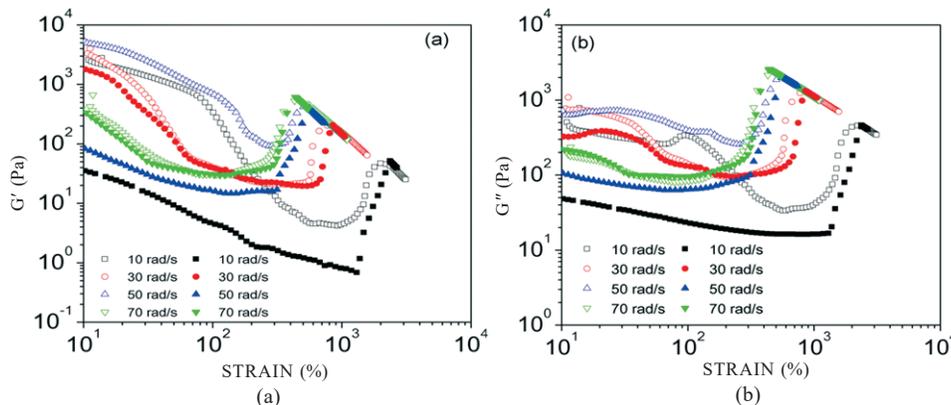


Figure 5. Comparison of (a) storage modulus and (b) loss modulus at different frequencies for 5 phr nanomer (open symbols) and fumed silica (close symbols).

show a striking non-linear (suspension like) response as the strain deformation is increased. High frequency and low frequency tests corresponds to rate of deformation same as would be seen at faster impact and slower impact at very high deformation time, respectively.

4. CONCLUSIONS

The present study demonstrates the development of relatively inexpensive STF of fumed silica-PEG with nanomer clay as additive dispersed at nanoscale. Clay is relatively cheaper than silica and is also ecofriendly. These factors will remain one of the primary advantages for using clay based additives over silica and other particles in shear thickening fluids in future. From the steady-shear experiments, it can be concluded that the addition of clay at low concentrations does not change critical shear rate and one of the advantages of clay is that high strains are typically sustained for much longer times as it takes time for many damage mechanisms due to impact to develop. Also importantly, clay makes STF less sensitive to change in temperature. Clay as nano-filler gives the same value of critical viscosity in both the steady-state and dynamic state. In contrast to only fumed silica STF, complex viscosity and both moduli increases as frequency and concentration increases for clay as nano additive. Thus, nano-clay as an additive increases both viscous and elastic nature. Most importantly, clay as an additive shows more stability and reproducible/consistent results. It was also established that the nano-particle shape matters and it is not simply the total percentage of solid particles in STF which determines its rheology.

Thus, the importance and advantage of using nano-clay as filler is clearly seen. It can be inferred that the amount of clay must be chosen judiciously to achieve the desired shear thickening behaviour. It will be interesting to study the effect on addition of various other nano-clays. Thus this novel study, incorporating clay as nanofillers, will lead to more research efforts in this direction.

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CONTRIBUTORS

Ms Mansi Singh received her BTech (Chemical Engineering) followed by MTech from IP University, New Delhi. All the experimental work has been carried out by Ms Mansi Singh in her capacity as JRF/SRF in the ARMREB project awarded

to Dr Rajeev Mehta. She is registered for the PhD under the supervision of Dr Rajeev Mehta. Her area of interest include polymers and specifically shear-thickening fluids.

In the current study, she carried out all the experimental work in her capacity as JRF/SRF in the ARMREB project awarded to Dr. Rajeev Mehta.

Dr Sanjeev K. Verma obtained his Masters and PhD from Himachal Pradesh University, Shimla. Presently he is working as Scientist 'D' with Ballistic Evaluation of Material group of – Terminal Ballistics Research Laboratory, Chandigarh. In the current study, he guided in synthesis of STF and analysis of data.

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In the current study she guided in analysis of data and execution of experiment.

Dr Rajeev Mehta received his BTech (Chemical Engineering) from IIT Delhi, MS from Virginia Tech, USA, and PhD from Thapar University. His main interest lies in the area of Polymers and polymer nanocomposites.

In the current study, his capacity as the Principle Investigator and the supervisor is very much involved with this research work- planning the experimental work and also analysis of the data, along with the conclusions arrived at.

Supplementary Figures

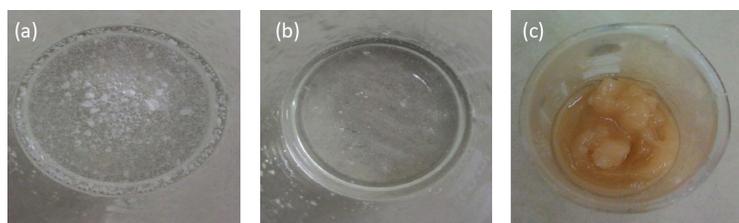


Figure S1. Fumed silica dispersed in PEG (a) without sonication; (b) with sonication (c) with clay as filler resulting in agglomerates due to improper mixing.

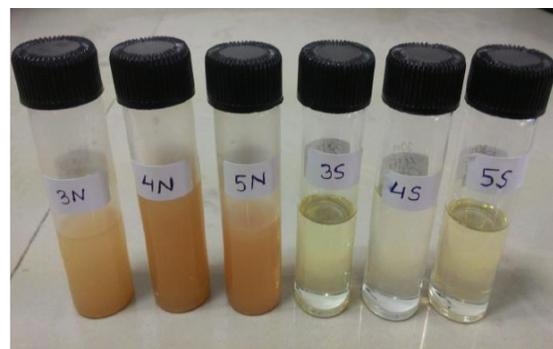


Figure S2. Glass vials containing STF samples.

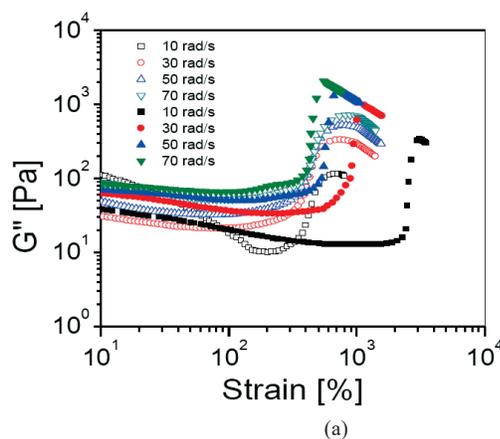
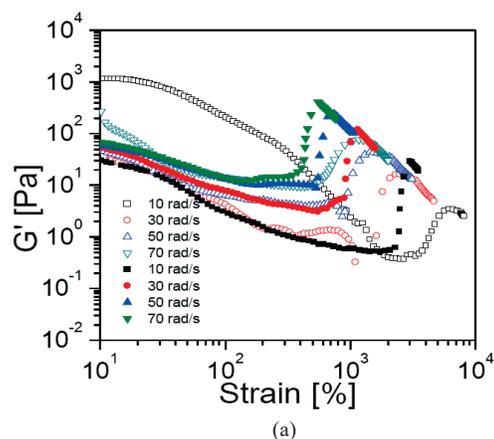


Figure S3. Comparison of (a) storage modulus and (b) loss modulus at different frequencies of 3phr nanomer (open symbols) and fumed silica (close symbols).