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Investigation of penetration into woven fabric specimens impregnated with shear thickening fluid

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Abstract: In this paper, the effect of weight fraction of nano silica (hydrophilic fumed silica particles) and molecular mass of polyethylene glycol (PEG) on the rheological properties such as the critical shear rate of fluids has been studied. Dynamic moduli based on strain and the effects of increasing the molecular weight are presented. Constructed samples with high-molecular-weight PEG have higher initial, final and critical viscosities. Also, higher molecular chains in the polymer and preventing the movement of most of these chains against the relative motion of liquid (viscosity) will cause higher viscosity in samples. Critical shear rate is lower in the provided samples with high-molecular-weight PEG. Polymer branches in these suspensions are absorbed by the surface of the particles. Due to OH bonds in the silica particles and also due to the presence of this bond in PEG, creating a hydrogen bond is likely. Such a hydrogen bond between the polymer yarn and the particle surface causes surface absorption of the particles. To show the effect of molecular weight on fibers, woven fabric specimens impregnated with shear thickening fluid (STF) have been examined by penetration and pressure test diagrams have been investigated. In a sample with higher molecular weight, displacement to yield point is higher and residence to penetration does not show much difference.

Keywords: dilution by ethanol; impact; nano silica; polyethylene glycol; shear thickening fluid (STF).

1 Introduction

The design of life-saving equipment such as flexible body protection systems critically depends on understanding the complicated non-linear dynamical response and

failure of advanced woven fabrics. The problem is challenging due to the interlacing weave of the fiber tows or yarns, the interaction between the projectile and the woven fabric and the effects of friction between various contacting entities [1].

The ballistic properties of a multilayered Kevlar fabric barrier may be improved with insignificant mass increase by impregnating it with shear thickening fluid (STF) [2]. STF is a composite material containing solid nanoparticles embedded in a liquid polymer, with a persistence of mobility.

Shear thickening behavior occurs in most viscous colloid suspensions consisting of hard solid particles [3].

Lee and Wagner [4], Lee et al. [2], Wetzel et al. [5] and Egres et al. [6] studied STF/fabric composites for several years. These studies reported the ballistic performances of composite materials composed of Kevlar fabric impregnated with a colloidal STF (micron-sized silica particles dispersed in ethylene glycol). The impregnated Kevlar fabric yields a flexible yet penetration-resistant composite material. Ballistic penetration measurements have demonstrated a significant improvement due to adding STF to the fabric without any loss in material flexibility. Such an enhancement in performance has been attributed to the increase in yarn pullout force upon transition of the STF to its rigid state during impact.

Polyethylene glycol (PEG) has an OH bond, and as SiO_2 also has OH bonds, hydrogen bonds will form between polymer yarns and particles [7]. The bridging process of polymers between particles will increase through increasing the fluid molecular weight, increasing particle-polymer flocs [8].

Because of longer polymer chains, the bridging process of the polymer will be simpler. Furthermore, bigger flocs will form and, naturally, lower energy is needed to bring the flocs closer and connect them together (forming hydro-clusters is simpler); therefore, a lower shear rate is needed to start shear thickening. Besides this, due to longer molecular chains in particles with fixed size, the chain bond to particles is weaker, and it is natural that the constancy of the suspension be lower [9–13].

Increasing the slope of viscosity is an important factor for STFs. The higher this slope, the higher the discontinuity in the increasing viscosity, which leads to higher

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energy absorption in the shear thickening area in the fluid and higher energy absorption from the penetrating foreign object to the composite [14–16].

In this paper, dynamic moduli based on strain and the effects of increasing the molecular weight are presented. Diagrams of pressure test have been investigated which is impacted by a cylindrical projectile.

2 Materials and methods

2.1 Materials and instruments

STFs were made by dispersing commercially available hydrophilic fumed silica particles (11–14 nm) at weight fractions of approximately 15%, 30% and 40% in PEG (with molecular weights 200 and 400), respectively (Merck Co.). To impregnate the woven fibers with the STF, the fluid should first be diluted. Therefore, 99% ethanol was used. One type of Kevlar fabric with 600 denier yarn and an areal density of 0.27 g cm^{-2} was tested. A scanning electron microscope (SEM) image of the silica nanoparticles is shown in Figure 1.

To make the STF, the polymer and nanoparticles are mixed together slowly. After adding a little of the nanoparticles to the polymer, with a mechanical blender, the force that is required for the mixture of the materials is created (see Figure 2A). For a better mixture, several blades are used. Each blade creates a different flow with a different speed in the fluid. This stage is continued until all the nanoparticles are distributed in the polymer. By means of an ultrasonic homogenizer, the

nanoparticles are distributed in the polymer completely. Sonic vacuum is the most effective method in providing and distributing. This method includes alternative sonic pressure that makes a hole in the liquid. In this study, a suitable ultrasonic homogenizer (Bandelin 3200, Germany) is used to disperse and prevent agglomeration (Figure 2B).

For both PEG 200 and PEG 400, 15, 30 and 40 wt% samples were prepared by dispersing the particles into the polymer using a mechanical mixer rotating at 1450 rpm. Mixing continued until a homogeneous and stable suspension was obtained. After preparing the suspension, it was left undisturbed at room temperature for 24 h in order to expel the air bubbles. Shear thickening fluid made by PEG and the silica nanoparticles have been shown in Figure 3.

2.2 Impregnating the woven fibers

To fabricate the STF-fabric composites, the STF was first diluted in ethanol at a 3:1 volume ratio of ethanol and STF. Individual fabric layers, each measuring $25 \text{ cm} \times 25 \text{ cm}$, were then impregnated with the solution for 1 min, squeezed to remove excess fluid, and dried at 60°C – 80°C for 20 min to remove the added ethanol and form the composite Kevlar-STF, as shown in Figure 4.

Then, three dried layers were positioned on each other. The STF weight additions reported for each target represented an average value over all of the target layers. Rheology analysis was performed to define how the STF's viscosity changes. Fluids were examined by geometry tests through a Searle type rotational rheometer. Strain sweep tests were performed for samples and dynamic moduli were obtained based on frequency. SEM analyses were performed in the central lab of Amirkabir University of Technology to define how the STF distributes among the fibers. Firstly, samples were sputter-coated with 30-nm gold and then photographs were taken using a Seron Technology SEM. Figure 5 shows SEM devices.

As shown in Figures 6 and 8 among the Kevlar-SiO₂ samples without diluting with ethanol, the upper fibers were connected properly to each other, but this was less observed in the intermediate fibers.

As shown in Figure 7 among the samples diluted with ethanol, there were some parts of the fibers that were covered by the STF, and the fibers were separated from each other and fewer bonds could be seen among the fibers by the STF. Figure 8 indicates the equal distribution of fluid and the particles on the surface of the fabric for the sample that included 40% silica particles in PEG.

Figure 1: SEM image of the silica nanoparticles.

Figure 2: (A) Mechanical blender; (B) Ultrasonic homogenizer.

Figure 3: Shear thickening fluid made by PEG and the silica nanoparticles.

3 Results and discussion

The viscous modulus indicates liquid-like behavior of the suspension, whereas the elasticity modulus indicates solid-like behavior of the suspension. Figure 9 indicates the dynamic moduli based on strain for samples with 15 wt% silica nanoparticles. Modulus measurement is performed at a fixed frequency of 10 s^{-1} and variable strain. In the sample containing PEG 200, the viscous modulus G'' is higher than the elastic modulus G' in all strains, which means that fluid viscosity overcomes the material elastic

Figure 4: Evaporation of ethanol from the impregnated fibers.

state and defines the constancy of the suspension. In the sample containing PEG 400, at strains lower than 30%, the G' elastic modulus curve is higher than the G'' viscous modulus curve. The viscous modulus indicates liquid-like behavior of the suspension, whereas the elastic modulus presents solid-like behavior the suspension. Therefore, this sample has an elastic behavior at low strains. On increasing the strain and transitioning from 30% strain, the curve of the viscous modulus is higher than the elastic modulus curve, which means that the sample has become steady.

From Figure 10, in the sample with PEG 400, the curve of the elasticity modulus G' is higher than the viscous G'' curve at low strains. The sample has a solid-like behavior at low strains (overcoming the elastic state on the viscous and solid-like behaviors of the suspension means that the fluid is inconstant). On increasing the strain and

Figure 5: (A) Sputter coater (B) Scanning electronic microscope.

Figure 6: Kevlar-SiO₂ samples without ethanol dilution.

transitioning from 35% strain, the viscous modulus curve is higher than the elastic modulus curve, which means that the sample has become constant at higher strains. Between 35% and 700% strain, the slope of the G' curve will reduce with a much greater intensity than the G'' curve. It means that on increasing the strain, not only are new hydro-cluster structures not formed, but the sample also moves forward to be more viscous and few structures in it will be destroyed.

In Figure 11, the sample consists of 40 wt% silica nanoparticles in PEG 400 and has an elastic behavior at low strains. On increasing the strain and transitioning

from 85% strain, the viscous modulus curve G'' will be higher than the elastic modulus G' , which means that the fluid viscosity overcomes the material elastic state and expresses the stability of the suspension.

In spite of samples with weight fractions of 15% and 30%, in the sample of PEG 200 with 40 wt% silica nanoparticles, it can be observed that at low strains, the elastic modulus is slightly higher than the viscous modulus, which means that fluid stability is reduced in this sample compared with the samples with a lower weight percentage. On comparing the curves in Figures 9–11, it can be observed that by increasing the fluid molecular weight and changing PEG 200 to PEG 400, the desired suspension is more unstable, which results from long molecular chains, making it ready for polymer bridging between particles and forming flocs in the suspension (flocculation of suspension), transitioning from a viscous state to an elastic state.

The characteristic of a fluid elastic state is a greater ability for load transition, which in the samples included more flocs. Due to more bonds between the polymer and the particles, load transition is higher and waste modulus (viscous modulus) is lower [9–13, 17].

Increasing the elastic and viscous moduli at high strains is due to shear thickening behavior. In Figures 9–11, for higher molecular weight samples at a lower strain percentage (lower initial force), shear thickening will begin.

In Figures 12–14, the obtained pressure test diagrams are presented and the effect of increasing the molecular weight is investigated. The samples have 15%, 30% and 40% of silica particles.

Figure 7: Kevlar-SiO₂ samples with ethanol dilution to scale of (A) 3 μm and (B) 30 μm.

Figure 8: Scanning electronic microscope photograph of the sample with 40% of silica particles in polyethylene glycol.

The square fabrics are placed as a single layer between two steel frames with 200-mm sides and 40-mm thickness. The diameter of the interior circle in the middle of the two metal plates is 127 mm.

In this quasi-static test, to exert a concentrated load on the surface of a 250 mm × 250 mm square fabric, a steel round-edged indenter with a cylindrical diameter of 12.7 mm and an edge length of 25 mm has been used. The concentrated force test has been performed at a speed of 6 mm min⁻¹. The test has been repeated three times for each sample.

From Figure 12, it can be observed that for samples with PEG 400, the most tolerated load is 2620 N and higher displacement is 49.2 mm until yield point. Also,

Figure 9: Modulus diagram based on strain percentage for the 15 wt% silica sample in (A) PEG 200 and (B) PEG 400.

for samples with PEG 200, the most tolerated load is 1852 N and higher displacement is 52.8 mm to yield point.

As can be observed in these samples, increasing the fluid molecular weight causes about a 29% increase in the load needed to yield and the displacement also increases

Figure 10: Modulus diagram based on strain percentage for the 30 wt% silica sample in (A) PEG 200 and (B) PEG 400.

Figure 11: Modulus diagram based on strain percentage for the 40 wt% silica sample in (A) PEG 200 and (B) PEG 400.

Figure 12: Load-displacement curve of the 15 wt% silica samples in PEG 200 and PEG 400.

Figure 13: Load-displacement curve for the 30 wt% silica samples in PEG 200 and PEG 400.

till failure point. In both these samples, compared to the raw sample, a noticeable increase can be observed in displacement and resistance against penetration.

From Figure 13, in sample with PEG 400, it is observed that the highest displacement to yield point is 34.4 mm and 32.6 mm for the sample with PEG 200. The most tolerated load for the sample with PEG 400 is equal to 3407 N and 3320 N for the sample with PEG 200.

From Figure 14, in the sample with PEG 400, it is observed that the highest displacement to yield point is 45.4 mm and 38.3 mm for the sample with PEG 200. The most tolerated load for the sample with PEG 400 is equal to 3604 N and 3552 N for the sample with PEG 200.

Figure 14: Load-displacement curve for the 40 wt% silica in PEG 200 and PEG 400.

Table 1: Results of the most penetrated force (n), maximum load to yield point (n) and maximum displacement to yield point (mm) for raw samples of 15%, 30% and 40% (PEG 200 and PEG 400).

Maximum force (n)	Maximum transition to yield point (mm)	Samples
1023 ± 22	44.4 ± 4	Neat Kevlar
1852 ± 19	52.8 ± 2	15% – PEG 200
2620 ± 28	49.2 ± 3	15% – PEG 400
3320 ± 22	32.6 ± 3	30% – PEG 200
3407 ± 36	34.4 ± 3	30% – PEG 400
3552 ± 31	38.3 ± 3	40% – PEG 200
3604 ± 41	45.4 ± 4	40% – PEG 400

As can be observed, in the sample with higher molecular weight, displacement to yield point is higher and residence to penetration does not show much disagreement. Results of the load-displacement curves are presented in Table 1.

From Table 1, it is observed that during the entrance of the penetrator to the fabric, a retarding force by the fabric is applied on the penetrator and the strain motions in the impact region along the fibers are transferred towards the edge of the fabric. Due to a lack of friction between the penetrator and the fiber, the presented fibers in the raw fabric in the region of the fibers' intersection slip and the penetrator passes through the fibers by pushing them. When a force is applied on a fabric surface saturated with the STF, nanoparticles that exist on the surface of the fabric accumulate at the impact point, and with increasing friction, prevent the slippage of fibers. Nanoparticles present in the thickening fluid can have an important role in increasing friction by being placed between the fabric and the pores in the fabric instead of the intersection of two strings. The penetrator in the sample saturated with the thickening fluid faces increasing viscosity during the application of force, which causes a decline in the

slippage of filaments existing in the fibers and lower slippage of fibers, further causing their tear, and then passes through the surface of the fabric. This leads to a higher loss of energy in the penetrator.

On investigating the results of the samples with PEG (Table 1), it can be observed that in samples that included 15% and 30% of the particles, increasing the molecular weight of PEG increases the residence to penetration of the fiber because on decreasing the molecular weight of PEG, the viscosity of the STF declines and the friction between all the parts decreases.

In the samples that included 40% of the particles, due to higher viscosity in both the samples, change in molecular weight has a slight effect on changing the resistance to penetration of the composite, and the change in yield point resulting from increasing the molecular weight compared with the samples with lower weight percentage of the particles is negligible in both the samples.

Resistance to penetration of the composite sample to the STF containing 40% of the particles indicates about a 3-fold growth compared with the raw sample. Also, energy adsorption by the composite in comparison with the raw fabric had a 2- to 3-fold increase, which resulted from energy adsorption in the shear thickening area. Furthermore, increasing the friction between the fibers, which leads to the addition of the STF into the fabric, is also a factor for energy adsorption.

4 Conclusion

According to the rheology analysis, increasing the molecular weight of the fluid causes increase in suspension inconstancy.

According to Figure 12, the suspension with the higher molecular weight fluid is more unstable, and with a low percentage of particles, higher molecular weight creates higher resistance to penetration.

The rheology results show that increasing the molecular weight of the fluid causes an increase in suspension viscosity, reducing the critical shear rate.

Pressure tests show that increasing the molecular weight of the fluid in samples containing 15% and 30% of the particles causes increase in resistance to penetration of the fabric, but in the sample with 40% of the particles, resistance to penetration does not change by increasing the molecular weight.

The smooth and relatively linear slopes of moduli in the thinning range of the constructed samples with a combination of mechanical and ultrasonic mixer indicate

that agglomerated particles do not exist in the fluid until micro scale and this is reflected in very good uniformity and stability of the structure. Thus, it can be concluded that although the mixing process and mechanical homogenization is relatively affective in lower weight fractions, using an ultrasonic mixer improves the stability of the suspension structure in both lower and higher weight fractions.

Resistance to penetration of the composite sample to the STF containing 40% of the particles indicates about a 3-fold growth compared with the raw sample.

References

- [1] Rao MP, Nilakantan G, Keefe M, Powers BM, Bogetti TA. *J. Compo. Mater.* 2009, 43, 445–467.
- [2] Lee YS, Wetzel ED, Wagner NJ. *J. Mater. Sci.* 2003, 38, 2825–2833.
- [3] Barnes HA, Hutton JF, Walters K. *An Introduction to Rheology*. Elsevier Science Publishers: Amsterdam, New York, 1989.
- [4] Lee YS, Wagner NJ. *Rheol. Acta.* 2003, 42, 199–208.
- [5] Wetzel ED, Lee YS, Egres RG, Kirkwood KM, Kirkwood JE, Wagner NJ. *The Effect of Rheological Parameters on the Ballistic Properties of Shear Thickening Fluid (STF) KEVLAR® Composites*. Proceedings of NUMIFORM, 2004.
- [6] Egres RG, Lee YS, Kirkwood JE, Kirkwood KM, Wetzel ED, Wagner NJ. *Novel Flexible Body Armor Utilizing Shear Thickening Fluid Composites*. Proceedings of the 14th International Conference on Composite Materials, July 2003.
- [7] Clements FE, Mahfuz H. *Enhancing The Stab Resistance of Flexible Body Armor Using Functionalized SiO₂ Nanoparticles*. 16th International Conference on Composite Materials. Kyoto, Japan, 2007.
- [8] Kamibayashi M, Ogura H, Otsubo Y. *J. Colloid Interf. Sci.* 2008, 321, 294–301.
- [9] Otsubo Y. *J. Colloid Interf. Sci.* 1999, 215, 99–105.
- [10] Auroy P, Auvray L, Linger L. *J. Colloid Interf. Sci.* 1992, 150, 893–899.
- [11] Napper DH. *J. Colloid Interf. Sci.* 1977, 58, 390–407.
- [12] Stenkamp VS, Berg JC. *Langmuir* 1997, 13, 3827–3832.
- [13] Zhulina EB, Borisov OV, Primitsyn VA. *J. Colloid Interf. Sci.* 1990, 137, 2.
- [14] Bettin G. *Energy absorption of Reticulated Foams Filled With Shear Thickening Silica Suspensions*. Massachusetts Institute of Technology, 2005, 92–98. Available at: hdl.handle.net/1721.1/33904.
- [15] Kordani N, Sadough A. *Sci. Eng. Compos. Mater.* 2014, 21, 421–425.
- [16] Kordani N, Sadough A. *J. Mech. Sci. Technol.* 2014, 28, 1–5.
- [17] Hai-lin Y, Jian-ming R, Zhong-cheng Z, Jian-peng Z, Qiu-mei W, Yuan-yan X. *J. Cent. South Univ. Technol.* 2009, 16, 926–930.