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The effect of carbide particle additives on rheology of shear thickening fluids

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In this paper, shear thickening fluids (STFs) including silicon carbide particles are presented. We fabricated a kind of STF based on nanosize fumed silica suspended in a liquid medium, polyethylene glycol, at a constant concentration of 20 wt.%. Then, different particle size silicon carbide (SiC) particles were added to the STF with various amounts. Their rheological properties under various temperatures were tested by using a rheometer. The suspension exhibits different systematic variations with respect to the varied parameters.

Keywords: shear thickening fluid, silicon carbide, additive, viscosity, shear rate

1. Introduction

Shear thickening fluids (STFs) are non-Newtonian fluids which have increased viscosity profiles under applied stresses. The viscosity turns to initial lower values by removing the stress from the medium. Hoffman (1972) made the pioneering study about thickening mechanism of STFs. It was stated that below a critical shear rate, particles in the suspension are in a hexagonally packed order. Beyond this point, particle formation decays thereby packed particles disorder and aggregate. This transition from order to disorder causes extreme increase in the suspension viscosity. Later, particle interactions in STFs were discussed by hydro-cluster theory (Wagner and Brady, 2009; Maranzano and Wagner, 2002). According to this theory, particles are contacted with each other under high stresses thereby increased hydrodynamic forces are acted inside the suspension. Consequently, aggregation of particles namely hydro-clusters, are formed and cause increased viscosity and jammed behavior of the fluids. Hydro-cluster theory was verified via simulations of Stokesian dynamics by Bossis and Brady (1989) and computational simulations by Boersma *et al.* (1992) and Melrose *et al.* (Silbert *et al.*, 1999; Catherall *et al.*, 2000; Farr *et al.*, 1997).

There has been significant interest for STFs to use their thickening behavior in the engineering applications such as body armor systems, damping devices, and smart structures. Body armor application is the most popular subject among the STF study areas. There are different types of armor systems such as STF impregnated fabrics, bulk STFs in containers and hybrid of them. Gates (1968) studied the first STF application in armor systems. After a long time, the subject has attracted much attention from researchers in the last decade. Previous studies focused on the STF impregnated ballistic fabrics to enhance the protection performance while reducing the weight of body armor (Majumdar *et al.*, 2013; 2014; Park *et al.*, 2012a; 2012b; Kalman *et al.*, 2009; Park *et al.*, 2014; Srivastava *et al.*, 2011; Laha and Majumdar, 2016; Tan *et al.*, 2005; Lee *et al.*, 2003). Contribution of STFs to the protection performance of ballistic fabrics is found as the common point in these studies. Petel *et al.* (Petel *et al.*, 2013; Petel *et al.*, 2015) investigated the ballistic performance of bulk STFs. It was expressed that particle hardness is a key factor on the protective performance of bulk suspensions. As the components of damping devices, STFs were used in the viscoelastic dampers to control the vibration of a structural part (Jolly and Bender, 2006). Increased profile of the viscosity was described as a damping behavior in case of earthquake or severe wind conditions (Seshimo, 1986). Zhang *et al.* (2008b) investigated the dynamic performance of an STF filled damper. Fischer *et al.* (2006) demonstrated the potential for integrating STFs into structures exposed to dynamic flexural deformation by controlling their vibrational response. Laun (1991) and Helber *et al.* (1990) studied the damping behavior of the STFs and indicated the application of STFs in the damping and mounting of the industrial machinery. STFs have also been studied in the applications of smart structures. Chu *et al.* (2014) verified the improvement of shear thickening effect by modifying the surface chemistry of silica particles. It was suggested that control of the rheology of colloidal suspensions provides important benefits in the applications of energy absorption materials. Hunt *et al.* (1991) investigated the minimization of damage in the equipment of controlled pulse fracturing (CPF) device by using STFs. Another invention was declared that STFs could be used in the composite structures by tailoring the stiffness via two elements having relative motions while covering the STFs. These composite structures were said

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to be applied for sports equipment, aeronautics, aerospace, consumer goods or in any other suitable field. Williams *et al.* (2009) investigated STF based medical equipment to restrict the movement of human joints such as shoulder, knee, elbow, ankle, and hip to prevent the joint from sudden accelerations. Galindo-Rosales *et al.* (2015) developed environmentally friendly composites including micro-agglomerated cork sheets with a network of micro-channels filled with STF. These composites were investigated under low velocity impact and the mechanical properties of these structures exhibited combination of mechanical properties of the micro-agglomerated cork sheets and the thickening response of the STF inside the micro-channels.

Despite the several studies about the STF applications, there have been very limited investigations in the literature about the effect of additives inside the STFs. Zhang *et al.* (2008a; 2010), Li *et al.* (2014), and Peng *et al.* (2014) studied the hybrid of STFs and magnetorheological (MR) fluids. The new fluid was named magnetorheological shear thickening fluid (MRSTF) and rheological properties of MRSTFs were investigated. Experimental results exhibited that MRSTFs have features of both fluids but they are more prone to MR fluids. Beside the magnetic particles as the additives in STFs, Petel *et al.* (Petel *et al.*, 2013; Petel *et al.*, 2015) investigated the ballistic resistance of the bulk STFs including carbide particles. According to these studies, carbide particles change the thickening behavior of the STFs while improving the ballistic resistance by increasing the suspension density and the dynamic material strength of the fluids. Starting from this point of view, we attempt to investigate the influence of additive carbide particles due to their rigidity even under loading. Effects of additives on rheological behavior of the STFs were investigated in order to provide some insights into the response of STFs and control the thickening behavior of these smart fluids. Systematical variations in the characteristics of STFs with respect to the parameters will contribute to determine the appropriate utilization fields for these novel suspensions in the engineering applications. In the present study, silicon carbide (SiC) particles were used as the additives in the STFs. Suspension temperature, particle size of the additives and amount of the additives were varied to observe the influences of different parameters. It was expected to provide new experience for the STF applications by this novel approach.

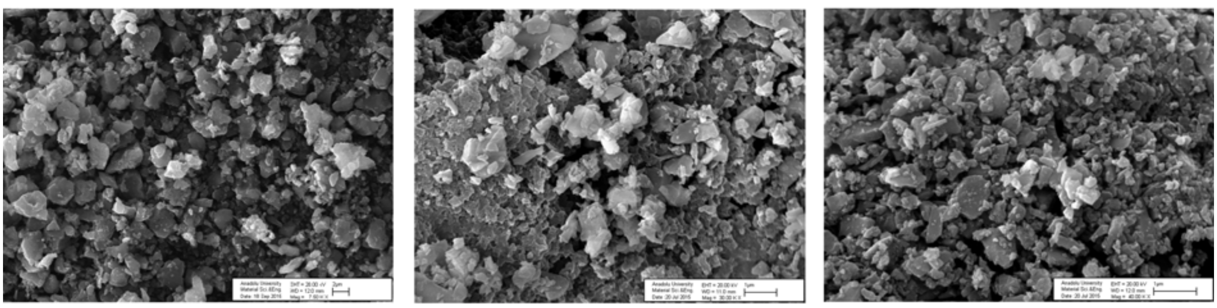
2. Experimental Details

The STF used in this study was based on fumed silica (Aerosil 90, from Evonik) which has primary particle size of 20 nm and specific surface area of 90 m^2/g. The liquid medium was polyethylene glycol (PEG) with molar mass of 400 g/mol (81172, from Sigma-Aldrich). Three different SiC particles, which were supplied from H.C. Starck Group, were used as additives in the STFs. Particle size and surface area measurements of SiC particles were performed with Zetasizer Nano ZS and Autosorb 1-C analyzer machines, respectively. Table 1 gives the measurement results of particle size and surface area. Fig. 1 shows SEM images of SiC particles which were taken with Zeiss Evo 50. It is obvious that each group of SiC particles has identical shapes which are irregular. Therefore, aspect ratio of the additives was remained stable and influence of the particle shapes was eliminated in the experiments.

Dispersion of the particles is important for dispersion quality and rheological properties of the nanocomposites (Galindo-Rosales *et al.*, 2011; Hussain, 2006). Although the energy input to the sample during the particle dispersion gives a rough estimate of the anticipated dispersion efficiency, methods with high energy per unit mass such as ultrasonic method are preferable in the sample preparation stage (Galindo-Rosales *et al.*, 2011). However,

Table 1. Particle size and surface area of SiC particles.

Additive code	Average particle size (μm)	Surface area (m^2/g)
SiC-A	1.114	7.61
SiC-B	0.512	14.39
SiC-C	0.340	27.20



(a) (b) (c)

Fig. 1. SEM images of (a) SiC-A at 7.5 K magnification, (b) SiC-B at 30 K magnification, and (c) SiC-C at 40 K magnification.

Table 2. Variable parameters and their levels.

Variable parameters		
Temperature (ºC)	Average particle size of additives (μm)	Amount of additives in STFs (%)
20	1.114	5
40	0.512	25
60	0.340	45

mechanical mixing is extensively used in the preparation of the STFs as in the previous studies (Petel *et al.*, 2015; Zhang *et al.*, 2008a; 2010; Tian *et al.*, 2015; Warren *et al.*, 2015; Gurnon and Wagner, 2015; Lee and Wagner, 2003). In the sample preparation stage, fumed silica was directly mixed with PEG. Silica loading was kept constant and used as weight percentage of 20% in the pure STFs. The mixture was mechanically blended for 40 min. After obtaining pure STFs, SiC particles were added to the suspensions according to the decided weight percentages. Then, the same mechanical blending procedure was applied to the mixtures.

In this study, effects of three parameters were investigated with three levels. Variable parameters were decided as suspension temperature, particle size of additives, and amount of additives in the STFs. Table 2 gives the variable parameters and their levels used in the experiments.

3. Rheological Testing and Results

Rheological tests were performed by using MCR 301 Anton Paar stress controlled rheometer with 20 mm diameter parallel plate apparatus. The gap between the plates was kept at 0.20 mm. Shear rate was increased from 0 to 1000 s^{-1} during the experiments. Suspension temperature was set with the help of a temperature control device connected to the bottom plate of the rheometer.

Before the experiments of suspensions with additives, effect of temperature on pure STFs was investigated. Therefore, pure STFs were tested at temperature of 20ºC, 40ºC and 60ºC, respectively. Fig. 2 shows the rheological curves of pure STFs with 20 wt.% silica loading under different temperatures. It is seen that temperature has four main influences on the characteristics of the STF. First, initial viscosity of the STF increases as the temperature decreases. In other words, viscosity curve profiles shift to upward on the graph by decreasing the temperature. Second, critical shear rate, which is defined as at which the shear thickening begins, decreases as the temperature decreases. Third, the thickening mechanism is formed more quickly if the temperature decreases. It can be also explained as the difference between the critical shear rate and the shear rate at the maximum viscosity reduces by decreasing the temperature. Last, thickening ratio (*TR*), which is defined in Eq. (1), increases when the temperature decreases. The rheological curves show that effects of temperature on pure STFs are consistent with the results stated in the early studies (Tian *et al.*, 2015; Warren *et al.*, 2015; Liu *et al.*, 2015). The mechanism of reduced viscosity at higher temperature can be defined by reduction in the strength of hydrogen links between silica and liquid medium. Furthermore, Brownian motion of the particles increases thereby structure is disordered by the effect of high temperature (Liu *et al.*, 2015). On the other hand, thickening takes place if the hydrodynamic lubrication forces overcome the inter-particle repulsive forces thereby stimulating the formation of the hydro-clusters. Nonetheless, inter-particle repulsive forces increase at the elevated temperatures and formation of the hydro-clusters requires larger shear rates. As a result, critical shear rate of the suspension increases at the higher temperatures (Liu *et al.*, 2015; Suh *et al.*, 2001; Kang *et al.*, 2012).

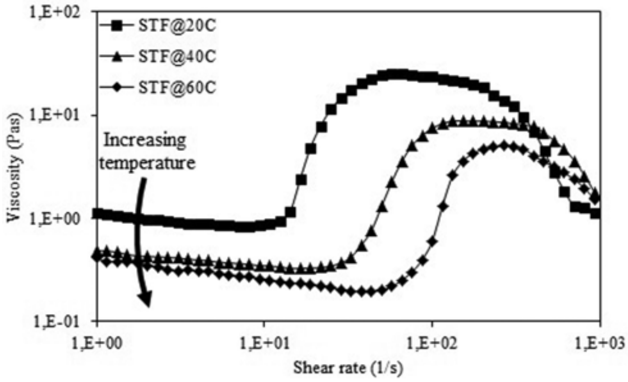


Fig. 2. Viscosity vs. shear rate curves of pure STFs at different temperatures.

$$TR = \frac{\eta_{\max}}{\eta_{cr}} \tag{1}$$

where $\eta_{\max}$ is maximum viscosity of the suspension beyond the thickening point and η_{cr} is viscosity of the suspension at the critical shear rate.

3.1. Effects on initial viscosity

In order to illustrate the effect of temperature on initial viscosity of STFs including additives, rheological curves of STF with 25% SiC-A at various temperatures were selected as shown in Fig. 3a. The other test results with constant particle sizes and amounts at different temperatures are consistent with these results. It can be stated that initial viscosity of suspension increases as temperature decreases. Temperature effect is obvious due to the weakening of inter-particle adhesion forces (Liu *et al.*, 2015; Nguyen *et al.*, 2007). Initial viscosity was also investigated by changing the amount of additives. The results are consistently obvious for each temperature and particle size

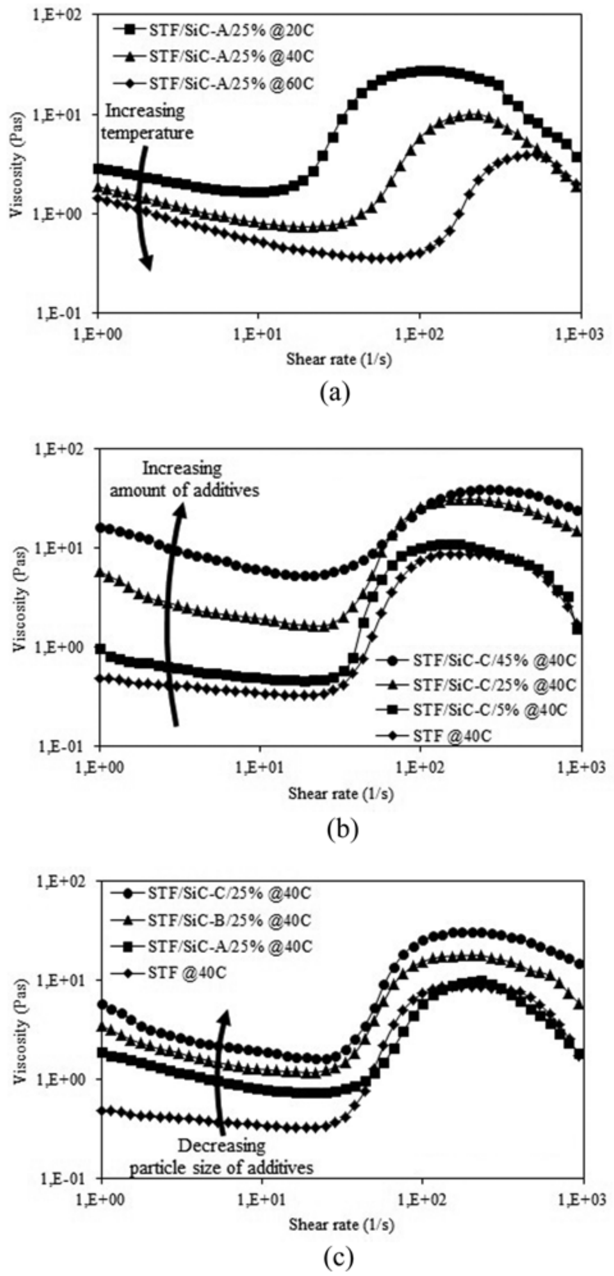


Fig. 3. Rheological curves of (a) STFs with 25% SiC-A at various temperatures, (b) STFs with various amounts of SiC-C at 40ºC, and (c) STFs including various particle size SiC with amount of 25% at 40ºC.

that if amount of additives increases in the suspension, initial viscosity increases. Fig. 3b shows the effect of additive amount by using rheological curves of STFs with various amounts of SiC-C at 40ºC. Increase in the initial viscosity can be explained by increased concentration of solid particles in the suspension thereby strengthened interparticle adhesion. It is known that viscosity is enhanced in denser suspensions (Liu, 2000). Upon increase of particle loading in the suspension, force networks are increasingly constrained and the viscosity increases (Mari *et al.*, 2014; Seto *et al.*, 2013). This effect can be also seen in the rheological results of the previous studies (Petel *et al.*, 2015; Zhang *et al.*, 2008a; 2010). According to the rheological tests in Petel *et al.*'s study (2015), initial viscosity of STF with 50.2 wt.% silica and 25.5 wt.% SiC loadings shows identical values with pure STF with 72.5 wt.% silica loading. Effect of particle size on initial viscosity of the suspension is shown in Fig. 3c. Rheological curves of STFs including various particle size SiC with amount of 25% at 40ºC were used to illustrate the results. According to the rheological curves, it is seen that initial viscosity of the suspension increases as the particle size of additives decreases. It is possible to mention that additives with finer particles are more effective to increase the initial viscosity of suspensions due to high number of particles distributed inside the suspension for the identical amount case.

3.2. Effects on critical shear rate

Fig. 4 shows the critical shear rates of STFs including various particle sizes and amounts of additives at different temperatures. There is overwhelming evidence that temperature has significant influence on critical shear rate values. For each particle size and amount of additives, critical shear rates give consistent trends with respect to the temperature. Critical shear rate of STF with additives decreases if temperature decreases similar to pure STF. Inter-particle repulsive forces increase at high temperatures. Therefore, higher hydrodynamic lubrication forces are required to cope with them. Consequently, critical shear rate of the suspension increases to attain higher hydrodynamic lubrication forces to form the hydro-clusters (Liu *et al.*, 2015; Suh *et al.*, 2001; Kang *et al.*, 2012). On the other hand, there is no systematic variation in the critical shear rate by the effect of particle size and amount of additives. However, STFs with higher particle amounts are more prone to increase the critical shear rates. This trend can be observed in the test results especially at temperature of 60ºC. Indeed, critical shear rate decreases if the concentration of silica increases in the suspension. This can be explained by sufficient number of silica in the medium to form hydro-clusters even if shear rate is at lower levels (Kang *et al.*, 2012; Barnes, 1999). Contrary to silica particles, SiC particles do not contribute to the thickening mechanism. According to Petel *et al.* (Petel *et al.*, 2013; Petel *et al.*, 2015), suspensions of ethylene glycol and SiC particles with various amounts exhibit shear thinning behavior rather than thickening. For this reason and the findings which will be described in further paragraphs, SiC particles negatively influence the thickening mechanism. Therefore, it is expected that critical shear rate increases by increasing the amount of additives. Although the results partially satisfy this expectation, we cannot point out a certain trend over

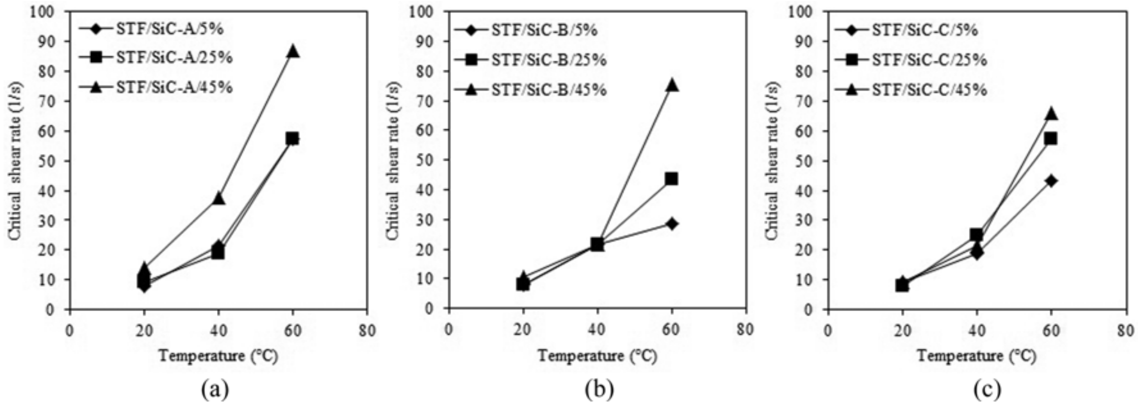


Fig. 4. Critical shear rate of STFs including (a) SiC-A, (b) SiC-B, and (c) SiC-C with various amounts at different temperatures.

amount of additives.

3.3. Effects on thickening period

We defined the thickening period as the difference between the critical shear rate and the shear rate at the maximum viscosity. Fig. 5 shows thickening periods of STFs including various particle size and amounts of additives at different temperatures. Effect of temperature on thickening period of STFs with additives exhibits identical behavior with pure STFs, which means it reduces by decreasing the temperature. Beside the influence of temperature, amount of additives effects the thickening period in a meaningful trend. Thickening period increases as the amount of additives increases as shown in the results. As stated in the previous studies (Zhang *et al.*, 2008a; 2010), additives deteriorate the thickening mechanism thereby completion of thickening is postponed to a higher shear rate. When additives are distributed in the STFs, additive particles intervene between the nanosize silica particles thereby obstructing the formation of hydro-clusters. Furthermore, number of obstacles increases by increasing the amount of additive particles in the suspensions which explains the test results.

3.4. Effects on thickening ratio

Fig. 6 shows thickening ratio of STFs with various particle sizes and amounts of additives at different temperatures. Results exhibit that thickening ratio changes inversely with temperature. It is known that to start up the thickening, hydrodynamic lubrication forces can easily overcome inter-particle repulsive forces at lower temperatures. However, it does not take place easily at higher temperatures since the repulsive forces are more predominant over the particles. Thus, it is possible to state that temperature has a negative influence on thickening ratio that restricts the thickening behavior of the suspension. Amount of additives has also significant effect on the thickening ratio. It is clear in the results that thickening ratio drastically decreases as the amount of additives increases in the suspension. In fact, additives act as disruptive agents for hydro-clusters by locating between the silica particles inside the STFs. Hence, they induce degradation of thickening mechanism so the thickening ratio by interstitial settling. There is a good agreement with the rheological results of Zhang *et al.* (2008a; 2010) in terms of thickening ratio. It can be seen in Zhang *et al.*'s study (2008a) that thickening ratio diminishes by increasing amount of

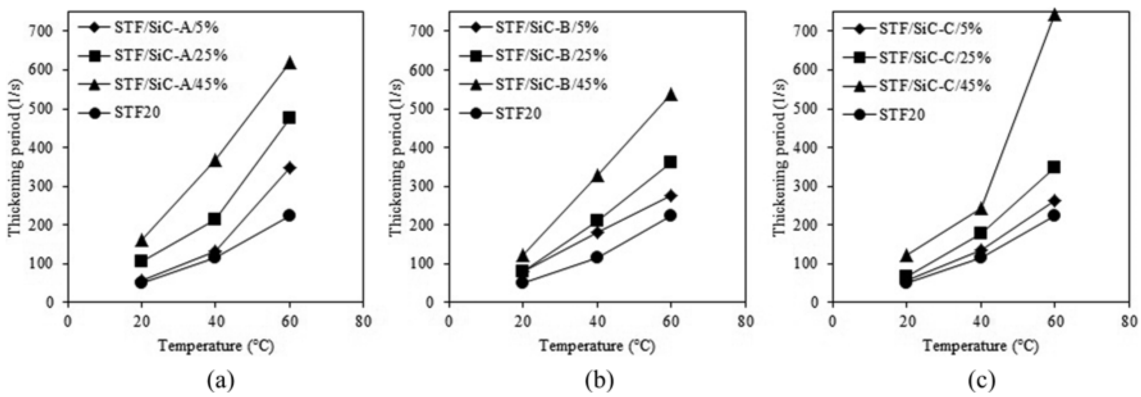


Fig. 5. Thickening period of STFs including (a) SiC-A, (b) SiC-B, and (c) SiC-C with various amounts at different temperatures.

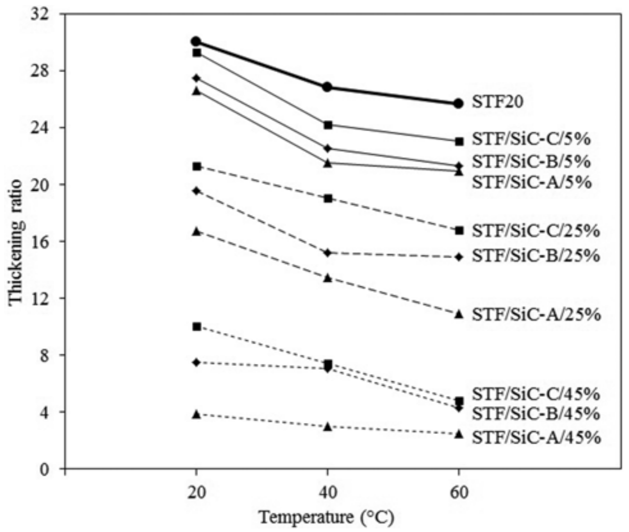


Fig. 6. Thickening ratio of STFs with various particle sizes and amounts of additives at different temperatures.

additives. Furthermore, thickening mechanism disappears at the maximum loading of additives in this study. It is also evident in Fig. 6 that additives with coarser particles have stronger effect on decay of the thickening in comparison with the finer particles. In the shear thickening, viscosity increases due to the higher number of finely dispersed hydro-clusters in the flowing medium. It is possible that additives with bigger particles constitute huge distances between the hydro-clusters in the flow field. However, as the particle size of additives decreases, size of the obstacles inside the suspension approaches to silica particles. Therefore, hydro-clusters can be less affected by smaller size of additives in the suspension.

4. Discussion

In the evaluation of the rheological results, four main characteristics of STFs such as initial viscosity, critical shear rate, thickening period and thickening ratio were investigated. Influence of each parameter on these characteristics was discussed, and summarized in Table 3. From the outcome of our investigation it is certain that additive of SiC particles inside the STFs disrupts the thickening mechanism of suspensions. This was verified by the diminishing trend of thickening ratio under all conditions. Disruption of the thickening mechanism can be also understood by observing thickening period which is another important characteristic of STFs exhibiting how quick the thickening works. In the results, thickening periods of pure STFs were shorter with respect to the STFs with additives. This finding can prove that response of the thickening mechanism decelerates by the effect of additives in the suspension thereby thickening mechanism is decayed. On the other hand, critical shear rate did not exhibit a systematic variation by changing the particle size and amount of additives. However, it had a significant increasing trend by the effect of increased temperature. Last, it is obvious that initial viscosity of STFs increases since additives are included in the suspension.

In recent studies, contact forces, rather than purely hydro-clustering approach, are commended to make contributions to shear thickening effects (Lin *et al.*, 2015; Mari *et al.*, 2014; Seto *et al.*, 2013). Denn *et al.* (Mari *et al.*, 2014; Seto *et al.*, 2013) reported that hydrodynamic interactions dominate the suspension at low shear rates due to the contactless rheology. However, contact forces are effective especially for the jamming point where the particles contact each other at high shear rates. It is also important that possibility of contacted microstructure increases as the concentration of solid particles increases in the suspension. Therefore, contact forces become stronger in dense suspensions even at the lower shear rates. It is stated that mildly dense suspensions are sufficiently dense for developing contact forces during the thickening (Mari *et al.*, 2014). Lin *et al.* (2015) performed shear reversal experiments to distinguish the relative contribution of the hydrodynamic and the contact forces in micro-size suspensions. Contact forces were claimed to be zero at the reversal stage of the experiments while assuming the microstructure was unchanged and the hydrodynamic forces were identical in magnitude but reversed in direction. In the light of this assumption, the qualitative difference between these forces was obtained to reveal the discrete contributions upon reversal. According to the

Table 3. Influence of parameters on characteristics of STF.

Increase in parameters	Trends on characteristics of STF		
	Increasing	Decreasing	No systematic effect
Temperature	Critical shear rate Thickening period	Initial viscosity Thickening ratio	
Particle size of additives		Initial viscosity Thickening ratio	Critical shear rate Thickening period
Amount of additives	Initial viscosity Thickening period	Thickening ratio	Critical shear rate

results, contact forces are more dominant in the thickening stage of the suspension. This approach was also studied for variable temperature and it was suggested that thickening onsets above a constant stress even the critical shear rate changes with the temperature. While shear reversal exhibits the effect of contact forces in the thickening mechanism, this model neglects the Brownian stresses which are inevitable for nanosize suspensions especially with lower concentration of solid particles.

According to the contact rheology model, additive particles do not contribute the thickening mechanism by breaking the silica particle contacts even the contact forces are expected to be increased due to the increased density of the suspension. This may be explained by the big difference in the particle size of the additives and the silica. Indeed, thickening behavior is less affected by smaller size of the additives. It may be possible that if the particle size of the additives approaches to the silica particles, thickening behavior could be evolved in a different manner. In order to validate this model, further studies are needed to be performed especially for nanosize systems in which Brownian stresses cannot be neglected. Furthermore, particle properties such as particle size and surface morphology should be investigated to observe the effect of contact forces. The fact remains that this model has been studied for single-phase suspensions so far and multi-phase suspensions are more complicated systems due to the interactions of different phases.

5. Conclusion

In this study, a novel approach for STFs was presented. SiC particles were used as the additives in the STFs and rheological behavior of these suspensions was investigated. Thickening mechanism of this new fluid exhibited systematic variations with respect to the different parameters. Additives provide an opportunity to modify the thickening stage of the STFs. Therefore, control of the thickening mechanism can be utilized in various fields. The findings suggest that this approach could be useful for the applications such as requiring higher initial viscosity and lower thickening ratio. Furthermore, additive particles can be employed to retard the activation of STFs by controlling thickening period. Controllable fluid actuators can be the main application area for this novel smart fluid.

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