Polymer suspension based shear thickening fluid fabric

Polymer suspension based shear thickening fluid-fabric for protective applications was a new idea. Shear thickening fluid was also known as STF. Generally STF contains liquid medium and solid particles which can be inorganic or organic based. Basically, the viscosity of the Shear Thickening Fluid (STF) will be increased when shear stress increasing. By exploiting this fluid deformation from STF, for the fabric itself, the friction between yarn-yarn and fiber-fiber will be increased drastically when force applied over the fabric composite. Then, the fabric itself will be having higher energy absorption when STF impregnated with the fabric itself compared with un-impregnated fabric composite.

The reason for this selection was motivated by previous works in this area, as well as the beneficial characteristics provided by these two materials. US Patents 5599290 and 5545128 have developed Bone fracture prevention garment and method”. In these two works, carried out by the same authors, the patent outlines a design used to resolve the issue of the harmful effects of impacts on the bones of elderly individuals. This design was described as being composed of the following: “ the component includes a dilatant material that was relatively stiff near the time of impact and relatively fluid at other times. In a preferred embodiment, the invention provides a hip pad, possessing a thickness small enough to be compatible with wearer acceptability, that conforms to the shape of the body during everyday activities such as walking, sitting, and sleeping, and was thus comfortable to the wearer” (5-14).

In order to model the rheological behavior of these fluids, it has been proposed to use a power law analysis with the relationship between the viscosity and shear rate represented by

Î· = k ýn-1

Where k is the fluid’s consistency and n is the power law exponent specific to the region of high or low viscosity. According to Barnes, the shear-thinning and shear-thickening regions can be accounted for by, “ using the sum of two power laws, with one value of n less than unity and one greater”. (8)

The rheology studied will be focused on colloidal suspension rather than hard particle suspension. Hard particle suspension like silica particle, was extensively studied by many researched over the world like Wagner and Lee Barnes (5 – 8) . They claimed, the hard particle suspended in the polyethylene glycol (carrier fluid) will exhibit shear thickening behavior by varying the volume fraction of the particle over carrier fluid from, above 40% to 55%. 30% by volume fraction will not exhibit shear thickening behavior but in Ragvahan experiments showed (10), by using 10% volume fraction of fumed silica suspended in the Polypropylene glycol (PPG), shear thickening behavior had occurred but likely referred to particle agglomeration(flocculated gel) rather than “ hydrocluster” because fumed silica, naturally have low surface area and larger particle size. In the experiment, the onset transition called critical shear rate seems having two points, the higher and the lower shear strain, due to the original structure of the carrier fluid itself (polypropylene glycol). The formation of vinyl group and straight chain in PPG gave two formations of clusters (10).

Colloidal suspension study was not emphasized and thoroughly studied because of the complexity of the colloidal system itself, which normally involved many factors such as steric and electrostatic stabilize, stability of the suspension and the formation of the hydrocluster via modification of the interaction particle of the colloidal suspension. In chapter 2, theoretical discussion about colloidal suspension which contributed to the new phenomenon of Shear Thickening Fluid (STF) such as structure-relationship of the colloidal suspension in polymer solution and Solid/Liquid transition (SLT) and also liquid/Solid transition (LST) [17]. According to the DVLO theory, stabilization of particle in the suspension with steric and electrostatic (will be have detailed explanation in the chapter 2) charges are important parameters to be investigated. The colloid particles were also known as surface charge particle. It involved the attraction and repulsive force for every single particle in the colloid dispersion or suspension, which contributed to the shear thickening behavior of the STF.

In this paper, colloidal suspension rheology will be main topics, by relating it with the structure-relationship and LST theory. In the chapter 2, more detailed onset transition of STF will be extensively discussed. Conventionally, the onset transition involved Order-Disorder transition (ODT) and hydrocluster.

The main criteria in Shear Thickening Fluid (STF) were to predict critical shear rate which was the critical point when the particles were suspended in the STF. It started to cluster with each other, exhibiting shear thickening behavior upon increase in shear rate. There were many factors in STF, some of them were, volume fraction dependence (between particle and carrier fluid), particle shape and size dependence (porosity) and particle interaction dependence. The particles can be normal charge (hard particle such as fumed silica, clay and any metal oxide particle) or colloidal particle (with charge).

In this paper, fumed silica will be used as a main material for hard particle as well as and colloid dispersion. Fumed silica will be turned into colloidal suspension by surrounding the charging over its surface particle. The main principal of colloidal suspension for this study was based on DVLO theory. Therefore, by studying the electrokinetic for each suspension, relation between this study with measured rheology through the power law index and yield stress value, there are possibilities to relate the structure- relationship in colloidal suspension of STF with onset transition (SLT & LST) evaluated. There were two basic requirements for exhibiting shear thickening behavior from colloidal suspension [raghavan, wagner, barnes], firstly, the volume fraction of the solid in the suspension must be very high and secondly, suspension must be nonflocculated or deflocculated

STF Fabric composite will be impregnated with STF has great potential in bullet proof application especially for soft armor. The vest will be having higher flexibility and less heavy than conventional soft armor. Wagner and his team claimed, STF will increase the friction between yarn-yarn and fiber-fiber by 500% using pull out yarn test. Also, by using NIJ as reference stabbed and puncture resistance test (NIJ 115. 00) for STF Fabric composite, it easily passed level 1 protection. For the bullet test, also NIJ as reference, it showed interesting results. Instead of higher penetration from the bullet (9mm) to the conventional fabric composite, for STF-Fabric composite, bullet was deflected away (rebound) and had less significant mark over the top of the clay in tested frame panel. Basically, fabric composite will be placed over the top of the clay. The mark of the clay will be used as reference for the dissipated energy (energy absorption) from the test. A bigger diameter of the mark and the depth for the mark was deeper indicating the fabric composite has a low dissipated energy system. Smaller diameter of the mark and less depth of the indented clay, showed, a higher dissipated energy system for the fabric composite.

1. 2 Problem Statement

Wagner found that STF had rheological characteristic of dilatant but studied on the use of hard particle in suspension. Colloidal suspension of fume silica in polymeric aqueous media was performed by other researcher but they studied only ionic strength and critical shear rate effect. In depth study on formation of hydroclusters in colloid need to evaluated to relate structure relationship between shear thickening behavior of polymer suspension with regard to composition (volume fraction), particle porosity, size and shape of particle. Hence, viscosity measurements were evaluated for different polymer systems to determine the effect of these systems on critical shear rate and shear thickening phenomena. Stabbed and puncture resistant fabric composite employing STF with high spike and knife impact performance can be affected by the formulation of STF and adhesion between STF suspension and fabric yarn. Hence, this study is hoped to solve the problems faced by indepth understanding on rheological and performance aspects in the development of STF fabric composite for protective applications.

1. 3 Objectives Of Study

The specific objectives of the project include:

- To determine physical colloid properties of colloidal suspension from hard sphere particles and colloidal dispersion via zeta potential studies.
- To determine the rheological behaviors of the colloidal suspension from the effects of repulsion system present during steady shear experiments.
- To fabricate STF fabric composite using various layers of Kevlar 49 and cotton fabric and determine stab and puncture resistance of STF fabric composite according to standard NIJ 115. 00 tests.

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CHAPTER TWO

LITERATURE REVIEW

2. 1 Shear Thickening Fluid (STF): Introduction & Review

Shear thickening fluid, basically have two different types of fluid behavior shear thinning and shear thickening. There are two types of shear thickening behavior in the fluid behavior. First, Shear thickening is a non-Newtonian flow behavior (dilatant) observed as an increase in viscosity with increasing shear rate or applied stress also known as time independent material (Barnes, 1989; Maranzano and Wagner, 2001; Lee and Wagner, 2003). Non-Newtonian flow behavior (rheopectics) observed as an increase in viscosity with increasing duration of stress (shear rate constant) known as the time dependent materials which have a memory to deform over period of time (figure 1).

Concentrated colloidal suspensions consisting of solid/hard spheres particles dispersed in a carrier liquid have been shown to exhibit rheological shear thickening behavior resulting in large, sometimes discontinuous increases in viscosity above a critical shear rate. This transition from a flowing liquid to a solid-like material is due to the formation of shear induced transient aggregates, or " hydroclusters," that dramatically increase the viscosity of the fluid (Barnes, 1989; Maranzano and Wagner, 2001; Lee and Wagner, 2003). For stabilize the STF and avoiding agglomeration behavior, co-solvent is added and it must be done because STF has a very strong molecule interaction between particles and carrier fluid (attraction force) rather than repulsive force when force is applied at specific time (critical shear rate and critical shear stress) [Barnes, 1989; Maranzano and Wagner, 2001; Lee and Wagner, 2003].

The Shear Thickening Fluid (STF) is the combination of the particles suspended in the carrier fluid. Figure 2 showed the STF curve when shear stress applied on the material. The particles used can be made of various materials, such as Silica Dioxide or other oxides, or polymers such as Polystyrene (PS) or Polymethyl methacrylate (PMMA), or other polymers from emulsion polymerization. Generally, particles can be in various shapes like spherical, elliptical, disk-like or clay particles (Barnes, 1989).

The particles can be stabilized in solution or dispersed by charge, Brownian motion, grafted polymers and others. Then, pH value of a suspension also contributed to the stability of suspension via colloidal condition such as steric repulsion and electrostatic system. The effects of pH value, concentration of the surfactant, and ionic strength of the surfactant are major factors for the colloidal suspension. This influence parameter is due to the agglomeration particle size (F. Rey, M. A. Ferreira et al. 1995). These are basic parameters in the rheological suspension and colloidal suspension system. Many studies of shear thickening fluid system (Barnes 1989, Hoffman 1998, Wagner 2004), focus on the colloidal particles only such as nanoparticles colloidal silica and monodisperse silica, which is it is well known as a very stable individual particle interaction in the suspension [Brownian suspension].

In this paper, electrostatic stabilize and steric repulsion system is used to control the interparticles interaction in the rheological suspension. Electrostatic stabilize is less studies due to the complexity in the rheological suspension. Concentration and ionic strength of the surfactant are the main parameter of this paper and the final pH value is critical measured in order to exhibit rheological shear thickening. F. Rey, and M. A. Ferreira in their paper " Effect of concentration, pH, and ionic strength on the viscosity of solutions of a soil fulvic acid" claims, all the parameter in this studies showed the dramatic instant result of the suspension due to the gel point of the suspension or well known as isoelectric point (iep) by adjusting H+ present in the rheological suspension. in addition of steric repulsion system, making a barriers for the individual surface particle is a intention in this paper. The double layers of the barriers (thickness) due to the zeta potential and electrokinetic theories are independent from the shear stress during rheological experiments. Because of the main intention of this paper are to determine the factors involving onset transition of the STF due to the interparticle interaction dependence which are closely related to the colloidal suspension rheology and the effects of the onset transition (rheological behavior) for the stab and puncture resistance test of fabric composite.

Then, the co-solvent that are used, it can be aqueous in nature and non-aqueous which can be chosen to stabilize suspension system The co-solvent should be environmentally stable like ethanol and methanol, so that, they remain integral to the fabric and suspended during service. Another function of co-solvent is to lower the viscosity of the STF, so that impregnation process of fabric composite becomes efficient and easy. By adjusting the viscosity of the STF, areal density of final fabric composite can be alter and also monitored. The particles must get through and suspended in the fiber-fiber and yarn-yarn [lee wagner et al 2003]. The result from the good impregnation process is the friction force between yarn-yarn and fiber-fiber will be increased [lee wagner et al 2003].

But in this paper, by using information from wagner and other researchers, a variation of the drying temperature for the STF and STF- fabric composite will be designed and tested via thermal degradation technique (pre-degradation) and swelling behavior of the STF-fabric composite after at the different drying temperature. The idea are to avoid pre-degradation region and swelling behavior of the STF in the drying process which is believed will reduced the performance of the fabric composite due to the following factors, degradation of the STF and STF-fabric composite due to the drying temperature and swelling behavior of STF. By combination those factors, believed, the internal friction between fiber-fiber and yarn-yarn will be reduced which is making inefficiency of energy absorbtion or dissipated energy for fabric composite (Wagner 2004).

2. 2 Mechanism of Shear Thickening Fluid (STF)

The mechanism of STF normally can be described via hydrocluster theory and order-disorder theory. The similiarity of these theories is a idea of a new macrostructure formation occurred, furthermore, it are closely related to rheological experiments such as temperature and time factors. A few researchers like Barnes (1989), Hoffman (1998) and wagner (2004), claimed colloidal factors such as different types of carrier fluid, particles porosity, and volume fraction of particle suspended. But, these two theories still can't explain in detail about the mechanism of STF. Wagner and his team claimed the formation of hydrocluster must be a deflocculated suspension and major parmaters of STF are volume fraction, interparticle interaction, and particle porosity. A new theoretical transition will be proposed for STF behavior. Liquid-Solid transition (LST) or semi-solid transition involving the relaxation state of the fluid during transition which is normally include the changes of loss and storage modulus during transition and believed new formation of a new macrostructure are formed during rheological experiment. This a new macrostructure formed due to the formation of the attraction force and repulsion force in the STF, which is believed closely related with double layer theories in zeta potential theory. LST theories is based on the transition of the material due to the changing of the complex modulus which are can be translated into the formation of gel (stiffness) and relaxation state of the material during near transiton and at the onset transition.

2. 2. 1 Hydrocluster

The most related theories to the shear thickening behavior are the hydrocluster theory. Basically, the hydrocluster will be occurred when the balance force from shearing flow in the concentrated suspension and the force rising from particle – particle (interparticles) interaction (Bender and Wagner 1995). There are two conditions, first via colloidal factors (steric and electrostatic repulsion) and particle interactions between particle-particle and particle-carrier fluid (Bender and Wagner 1995). Figure 3 showed the formation of hydrocluster, when the applied shear stress on the STF.

The formations of hydrocluster are still extensively investigated by researcher in the entire world. The main interest of this unique behavior is the onset transition (critical shear rate) from liquid state to solid or gel state. This phenomenon involving the rapid changes of fluid viscosity in a second whether applied it with shear stress, applied electric field (refer to the ER fluid), and applied magnetic field (refer to the MR fluid). There are specific equipments for detection of this phenomenon such as optical rheometer, small angle neutron scattering (SANS), and two beam light laser scattering. But with all this equipments, still the formation of hydrocluster is unknown phenomena.

According to this theory, at the lower stress (below critical stress, maximum volume packing fraction) interparticle interaction either Brownian motion or electrostatic, making the concentrated suspension is easily flow (shear thinning behavior and viscosity of the suspension become lower) due to the particles slippage in the carrier fluid. As the stress is increased, the attraction forces is slightly increases than repulsion force in between particles of the concentrated suspension (particles aggregation become larger and the viscosity of the suspension become higher). When the magnitudes of the shearing force are equilibrium to the interparticle interaction, the particles in the concentrated suspension become " cluster" also known as hydrocluster.

This theory is first suggested by Brady (Bossis and Brady 1989) as a result from stokesian Dynamics simulation and then, supported by optical rheological experiment by wagner (Bender and Wagner 1995). Basically, these phenomenons are closely related with phenomenon of " Resonance" in physic. In physics, resonance is the tendency of a system to oscillate at greater amplitude at some frequencies than at others. These are known as the system's resonant frequencies (or resonance frequencies). At these frequencies, even small periodic driving forces can produce large amplitude oscillations, because the system stores vibration energy [the fundamental of physic 1999].

2. 2. 2 Order-Disorder Theory

The order-Disorder mechanism is first suggested by Hoffman (Hoffman 1972) which obeserved the monodispersed supension under shear generate different patterns at critical shear rate (before and after). According to this theory, when the suspension is sheared, particles is ordered in the specific formation (hexagonal or pentagonal) of packed layers parallel to the plane of shear. After a crtical stress is reached, the instabilities in the formation of packed layers become greater and particles are out of the formation. Then, these particles collide and jammed to each other and produce in the rise of viscosity.

An example of these pattern can be seen in figure 4 Hoffman monitored the monodispersed suspension under shear and showed that figure 4(A) corresponds to the order formation of the packed layers while figure 4(B) is disorder or random formation of the packed layers after critical shear stress is reached.

The illustration of the phenomenon for the order- disorder formation of the packed layers that suggested by Hoffman can be seen in figure 5. those formation is captured by the Hoffman by using a simple shear in figure 4 (A) is same formation in the figure 5 (A). Hoffman predicted that, those formation occur due to the strong surface bonding in the monodisperse suspension which normally reffered to the hydrogen and van der waals bond. In figure 5 (B), the disorder formation occurs when sample shear above critical shear stress. Particle are collide and jammed each other and produce in rise of viscosity due to the agglomeration of the particles.

2. 2. 3 Introduction Liquid-Solid Transition (LST)

In this paper, a new concept theoretical onset transition of STF will be proposed. The main ideas of this theoretical concept are the macrostructure of the fluid are changes during onset transition from liquid state to the solid or gel state, and liquid- solid transition state which is normally involving the relaxation state (?????). The transition also can be state as semi-solid transition at the critical shear rate. This paper, also will be investigated the formation of the transition by using colloidal particles which are stabilize by using two system, steric and electrostatic repulsion system.

LST involves many such of factors, such as theory of gelation, branching theories, and percolation theories. All the theory in LST are closely related to the formation of the macrostructure, whether effect on the temperature surrounding or over time.

- 2. 2. 3. 1 Theory of Gelation

The LST of polymers is also technically important since it occurs in nearly all of the common fabrication processes. Examples are injection molding of semi-crystalline polymers (where the surface quality of the finished parts may be affected by gelation shear thickening fluid (STF) and processing of crosslinking polymers. Therefore, the onset transition for STF can be detected by using LST.

There are several theories in gelation are normally used in LST. First is branching theories and second is percolation theory. The onset transition are very important to the STF for comparison with the hydrocluster formation theories and order – disorder theory and proposed new theoretical idea for the rheological behavior due to the factors affecting the performance of STF such as molecular weight dependence, volume fraction dependence and particle – particle interaction dependence. Those all factors which affecting the performance of STF had been discussed in previous sub-chapter.

2. 2. 4 Description of the Phenomena for Shear Thickening Fluid (STF)

Basically, the phenomenon of STF is investigated by using a lot of parameter in the last two decades. Volume fraction, particle porosity and interparticle interaction dependence is a major parameters for STF. In this paper, molecular weight of carrier fluid is added in the STF's parameter, in order to increase the potential parameter for the STF's phenomenon.

- 2. 2. 4. 1 Volume Fraction Dependence

Volume fraction factor is the main parameter in the shear thickening fluid [STF]. In general, a solid or hard particle which is suspended in the carrier fluid such as ethylene glycol, polyethylene glycol or other carrier fluid which are aqueous in nature or non-aqueous in nature can exhibit shear thickening behavior at the minimal range of volume fraction in between 30% to 49%. Above 50% of volume fraction, the rheological shear thickening behavior can be measured at lower shear rate but it depending on the complex viscosity of the suspension which is it is related to another parameters like particle size and porosity. Meaning, surface area and aspect ratio of the particle are greater influenced on the final viscosity of the STF.

One parameter that has a huge effect on the critical shear rate is the volume fraction. At low volume fractions (below 0. 5), shear thickening is either less dramatic or not significant [Characterization of Shear-Thickening Fluid-Filled Foam system for Use in Energy Absorption Devices, Jose 2004].

Wagner and his team also claims, colloidal silica (14nm) which is suspended in the polyethylene glycol, less than or 30% volume fraction of STF will producing less or no significant of thickening behavior either at low shear rate or higher. Raghavan and khan which studying the rheological behavior of fumed silica suspended in low molecular weight polypropylene glycol claim, non-flocculated suspension exhibit shear thickening at 10% [w/w] under steady flow and strain-thickening under oscillatory shear. Strain-thickening refer to the abrupt increase in the complex modulus [complex viscosity].

Fumed silica generally known has higher agglomeration size up to 140 micron. Therefore, volume fraction has less significant affected on the shear thickening behavior. The agglomeration size is the main factor in the Raghavan and Khan studies (figure 2. 2. 4).

The results of Barnes demonstrate that at volume fractions in the range of 50 %, the shear thickening behavior is expected and predictable. In addition, theoretical analysis of the maximum volume fraction of monodispersed suspensions predicts this value is Ømax= 0. 605, where this value corresponds to, “ the volume fraction for a cubically stacked hexagonal packing” (Boersma et al. 1989).

- 2. 2. 4. 2 Molecular Weight of the Carrier Fluid Dependence

Molecular weight of the carrier fluid is a new parameter in the STF. Wagner and his team reported that the viscosity of the carrier fluid is important to predict the onset transition from shear thinning to the shear thickening behavior in the STF. A difficulty occurs from getting exacts experimental measurement of the effect of carrier fluids due to the reality that changing the carrier fluid affects the interparticles interaction. In this paper, by monitoring the zeta potential of the carrier fluid, those two effects (molecular weight dependence and Particle-Particle Interaction Dependence) can be separated.

The onset transition of STF will be a main indicator for this parameter neither the suspension are flocculated or deflocculated. The idea of used difference molecular weight is cames from colloidal suspension which prepared by raghvan, in his experiment polypropylene glycol (PPG) as a main carrier fluid. He are experimental the effect of the rheological behavior for fumed silica suspended in PPG at lower concentration (mass fraction), and showed the STF behavior at low yield stress (figure 6). The experiment by raghvan is difference from wagner and barnes, which used colloidal particle from Nissan Chemicals (MP4540) and suspended in PEG 200 at high concentration for exhibit shear thicknening behavior (figure 6).

Therefore, when turnable fumed silica particle into colloidal fumed silica via steric and electrostatic repulsion system, suspended it in the different molecular weight of carrier fluid at various particle loadings, believed, the suspension will exhibited shear thickening behavior at low yield stress.

- 2. 2. 4. 3 Particle-Particle Interaction Dependence

Interparticles interactions are very important in determining the shear thickening behavior of a suspension. Flocculated suspension will not exhibit shear thickening (Barnes 1989), but instead they will show shear thinning, as shown in figure 7.

Basically, the flow behavior of a suspension is extremely affected by interparticle interaction. These phenomenons also refer to the final condition which refers to the pH value of a suspension. Therefore in table 1, the Floc sizes are monitored due to the effect of pH value, electrolytes, and polymer on a kaolin suspension. Because of flocculation is expected to begin at lower pH values, namely edge to face that kaolin is positively charged and de-flocculation at higher pH values it carries a net negative charge on the surface. (Nongkhran Chaiwong 2008).

The flocculation of kaolin depended on pH, electrolytes and polymers flocculants. Floc size and floc strength increased with increasing of cation valency in the electrolytes and increasing of molecular weight in the polymers (Nongkhran Chaiwong 2008).

The information that gathered from table 1 is important for the colloidal suspension