



# Article Impact Resistant Flax Fiber Fabrics Using Shear Thickening Fluid

Joseph Fehrenbach, Eric Hall, Luke Gibbon, Tanner Smith, Ali Amiri 🗈 and Chad Ulven \*🖻

Mechanical Engineering Department, College of Engineering, North Dakota State University, NDSU Dept 2490, P.O. Box 6050, Fargo, ND 58108, USA

\* Correspondence: chad.ulven@ndsu.edu

**Abstract:** Shear thickening fluids (STFs) have been shown to improve the effectiveness of fabrics used in soft body armor applications. They are used to increase the puncture and ballistic impact resistance of Kevlar<sup>®</sup> fabrics. However, the effect of using STFs with natural fabrics such as flax appears to have never been studied. Similarly, the hybridization of different fabric types impregnated with STF has also only undergone limited study. The rheology of STFs at varying concentrations of nanosilica dispersed in polyethylene glycol (PEG) was studied at different temperatures. It was found that the STFs behave as a non-Newtonian fluid in response to changes in shear rate. In this study the effectiveness on the puncture and ballistic impact resistance of impregnating flax fabric with STF at concentrations of 30%, 50%, and 70% w/w of nanosilica in PEG was investigated. The effect of hybridization of flax and Kevlar<sup>®</sup> fabrics impregnated with STF was also investigated. The puncture resistance of both flax fabrics treated with STFs and hybrids treated with STFs was found to increase in the hybrid samples when STF concentration was at least 50%. The flax treated with STFs showed either a decrease in specific energy absorption per layer for the lower STF concentration, or a very small increase at 70% STF concentration.

**Keywords:** shear thickening fluid; natural fiber; flax fiber; composite; aramid; impact resistance; puncture resistance

## 1. Introduction

Shear thickening fluids (STFs) are dense colloidal suspensions that exhibit abrupt increases in viscosity with increasing shear rate [1]. STFs are produced by homogenously dispersing hard particles inside a Newtonian carrier liquid. Shear thickening behavior was initially seen as undesirable, because in mixing processes the effect can cause greater stress on equipment and energy demands in order to achieve mixing [2]. More recently the unique behavior of these non-Newtonian fluids has led to attempted development of smart materials and structures utilizing STFs.

Research concerning STFs has been completed in a variety of applications including body armor, structural damping, heavy machinery damping, and medical rehabilitation [3,4]. For most body armor applications, high performance aramids are used and offer improved flexibility and lower weights compared with traditional protective materials [5]. Impregnating fabric materials with an STF has been shown to significantly increase puncture and impact resistance of the material. This is attributed to an increase in the inter-yarn friction or yarn pullout force in the fabric material due to the increase in the STF's viscosity. Many researchers have investigated the effects of impregnating STFs into aramid fabrics, which has shown significant impact resistance [6–9].

There are several factors which can affect the rheological properties of STFs. In order for particle suspensions to show shear thickening behavior a minimum concentration of particles in suspension must be reached. The particle geometry can affect the rate at which



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). shear thickening is induced. In initial studies it was discovered that rod-shaped particles are the most effective at improving the shear thickening effect in the suspension [10,11]. Temperature also affects the rheological properties of an STF. Srivastava et al. [11] have studied the effect of temperature on the rheology of a PEG/silica STF. It was observed that at 0 °C the shear thickening effect is most pronounced, with least effect at 50 °C. Particle size and hardness also effect the performance of an STF. In general, smaller particles enhance the shear thickening effect more than larger particles. Harder particles are desired for their higher mechanical properties and ability to withstand higher shear stresses, although particle hardness has no direct effect on the STF performance.

A major influencer of the rheology of the STF is the particle volume or weight fraction. It is generally accepted that as the particle loading is increased, the shear thickening effect is enhanced. Wetzel et al. investigated the rheology of PEG/nanosilica suspensions at varying volume fractions [7]. It can be seen that the suspensions which exhibit shear thickening first show a region of shear thinning when the shear rate is low. As it increases, a critical shear rate is reached and the suspensions show abrupt increases in viscosity. Additionally, at higher particle volume fractions the critical shear rate required to induce the shear thickening is reduced.

STFs are produced by homogenously dispersing hard particles inside a Newtonian carrier liquid. Ultrasonication is among the popular methods used for dispersing particles to create STF. This process applies sound energy to agitate particles at a frequency >20 kHz. The cycles of sound wave pressure lead to the formation and subsequent cavitation of microscopic vacuum bubbles when used in solution, which aid greatly in mixing and degassing processes [12–15].

Due to their high impact and puncture resistance, impregnation of STFs has been most heavily studied in aramid fabrics. Through impregnation of nanosilica/PEG STF in an aramid fabric, an increase of 623% in ballistic impact absorption and a 500% increase puncture resistance was seen [16–25].

The hybridization of fabric materials impregnated with STFs has received limited study but could possibly lead to a material with a unique combination of properties. Harish Rao et al. studied the stab characterization of an aramid and ionomer thermoplastic film hybrid material impregnated with an STF. They measured significant increases in energy absorbed from dynamic stabbing impacts and also force required to puncture the material in quasi-static puncture tests [26]. Edison E. Haro et al. investigated ballistic impact response of a laminated hybrid composite consisting of an aluminum alloy, epoxy, and Kevlar<sup>®</sup> impregnated with an STF and found improved energy absorption [27].

Although effects of STF impregnation on synthetic fabrics has been studied, there is little information regarding its effects when impregnating natural fabrics such as flax. In terms of its mechanical properties, flax is considered to be one of the strongest natural fibers [28]. As such, flax is a promising candidate for study with impregnation of an STF. This is an area of limited study. Hybridization of flax fabric and aramids, both impregnated with an STF, could lead to a material suitable for use in body armor applications while being partially bio-based and less costly.

The goal of this work is to verify that STFs have a beneficial effect on flax fabric's puncture and ballistic impact resistance, as this has never before been studied. This is important because it could show that different types of fabric materials impregnated with STFs can be used to potentially create a new class of impact resistant materials. These materials could be fabricated from readily available plant-based fibers and silica particles, or even more readily available particles such as corn starch. It is hypothesized that a completely bio-based and totally renewable puncture and impact resistant material can be made via utilization of a bio-based particle suspension impregnated into bio-based fabrics.

#### 2. Materials and Methods

#### 2.1. Materials

A 2 × 2 twill weave flax fabric of areal density 0.412 kg/m<sup>2</sup> was used for the experiments and supplied by Composites Evolution, Chesterfield, UK. The flax fiber used in this fabric typically exhibits tensile modulus around 60 GPa and strength around 800 MPa. A plain weave Kevlar<sup>®</sup> 29 fabric of areal density 0.459 kg/m<sup>2</sup> was used for the experiments supplied by Fibre Glast Developments Corp, Brookville, OH, USA. Kevlar 29 fibers exhibit a tensile modulus of 70 GPa and strength of 2900 MPa.

Nanosilica or silicon dioxide nanoparticles, are an oxide of silicon with the chemical formula SiO<sub>2</sub>. Silica is a naturally occurring material commonly found in living organisms, quartz, and sand. It is abundant and is used in structural components, electronics, components in food, and also in the pharmaceutical industry. Nanosilica spheres of ~15–20 nm diameter were supplied by Nanostructured & Amorphous Materials, Inc, Katy, TX, USA.

PEG with a molecular weight of 200 was used for producing the STF. The chemical formula of PEG is  $C_2nH_4n+2On+1$ . The chemical structure of the monomeric unit of PEG is shown below in Figure 1. Ethanol of 95% concentration was used for diluting the STF in order to reduce its viscosity for impregnation of fabrics. The PEG and ethanol were purchased from Sigma-Aldrich, Burlington, MA, USA.



Figure 1. Chemical Structure of PEG.

#### 2.2. Processing

STF samples used for fabric impregnation were produced via a one pot method. STF constituent materials were first added into a container and subsequently diluted with ethanol. As the viscosity of the STF suspensions is too great to impregnate the fabrics, it must be diluted. In order to facilitate the impregnation, STF samples were produced in a single container at varying concentrations of nanosilica in PE; 30%, 50%, and 70% wt/wt. The samples were then diluted at a 1:3 volume ratio of STF to ethanol. The suspension was manually stirred to break up any large agglomerates and then sonicated at 600 W, 20 kHz for up to 10 min or until homogenous using pulses of sonication lasting 30 s with 30 s pauses. A MixSonix 3000 probe type sonicator manufactured by Misonix was used for ultrasonication-based dispersion of nanoparticles into the carrier fluid.

For puncture testing, 20 sections of 6.4 cm  $\times$  6.4 cm squares of both flax and Kevlar<sup>®</sup> fabric were cut for use. This was accomplished for each sample concentration. Likewise, 20 sections of 21.6 cm  $\times$  21.6 cm of both fabrics were cut for use in ballistic impact in each concentration. The weight of each layer of fabric was recorded prior to impregnation in order to measure the weight change post impregnation.

Once all STF samples were diluted and mixed, they were poured into a container. Each fabric layer of flax and Kevlar<sup>®</sup> was submersed in the desired suspension concentration for 120 s. Fabric layers were then removed from the suspension and placed in a convection oven at 80 °C for 30 min to evaporate the ethanol from the fabric. Samples were dried, the mass was recorded using the analytical balance, and areal density was calculated.

### 2.3. Testing

#### 2.3.1. Rheological Characterization of STF

An ARG2 rheometer was used to evaluate the rheological properties of the STF samples at 30%, 35%, 40%, 45%, and 50% w/w concentration of nanosilica in PEG. Rheological experiments were conducted under steady, shear-stress-controlled tests. The spindle used was a 40 mm diameter 2° cone with a gap distance of 58 µm. The rheometer was programmed to measure from shear stress of 0.1–2000 Pa collecting data at a frequency of 10 data points per decade. Tests were run at both 25 °C and 50 °C in order to analyze the effect of temperature on the rheological properties. Unfortunately, the rheological properties of STF samples above 50% w/w concentration of nanosilica in PEG could not be measured due to physical limitations of the equipment used.

#### 2.3.2. Puncture

An Instron 5567 electric load frame with a 30 kN load cell was used for conducting puncture testing. Puncture testing was conducted according to ASTM F1342 for Protective Clothing Material Resistance using Puncture Probe A [29].

For these tests, four layers of flax fabric, four layers of Kevlar<sup>®</sup> fabric, and a hybrid of two layers of flax and two layers of Kevlar<sup>®</sup> fabrics (layers: Flax/Kevlar<sup>®</sup>/Flax/Kevlar<sup>®</sup>) were impregnated with varying levels of STF suspension; 0, 30%, 50%, and 70% w/w concentration of nanosilica in PEG. The fabric layers were centered and clamped in the testing fixture. The steel spike was then moved to contact the specimen. The spike was then forced into the fabric at a rate of 50 mm/min until it punctured the fabric. The tests were run five times for each sample.

#### 2.3.3. Impact

A custom high velocity ballistic impact testing system was used for ballistic impact testing. inside the box, two chronographs are mounted where the specimen is mounted. The first records the input velocity before impact, while the second records output velocity after the projectile penetrates the specimen. The samples are clamped securely in between two steel plates with abrasive tape adhered to the inside clamp against the specimen in order to prevent slip during impact. The plates are then secured in place via a secondary clamping system. For flax fabric samples four layers of fabric were clamped in the device for ballistic impact testing. For hybrid samples two layers of fabric were clamped in the device for testing.

Projectiles used for the experiments were steel with a flat, blunt nose and were loaded into a sabot which fits snugly inside the barrel. The projectiles weighed approximately 17.0 g. Figure 2 shows the type of projectiles used in the ballistic experiments.



Figure 2. Projectile.

Again, four layers of flax fabric, four layers of Kevlar<sup>®</sup> fabric, and a hybrid of two layers of flax and two layers of Kevlar<sup>®</sup> fabrics (layers: Flax/Kevlar<sup>®</sup>/Flax/Kevlar<sup>®</sup>) were impregnated with varying levels of STF suspension; 0%, 30%, 50%, 70% w/w concentration of nanosilica in PEG. The four fabric layers were centered and clamped tightly between the steel frame. Steel projectiles were fired at the specimens and the maximum velocity at which the specimens were not punctured was recorded. Each sample type was tested with three specimens. The input velocity and mass recorded determined the energy absorbed during the impact using equations as there was no exit of the projectile.

## 3. Results and Discussion

## 3.1. Rheological Tetsing

The viscosity of the STF produced as described in Section 2.2 was tested as described in Section 2.3.1 to establish it was showing shear thickening behavior. Shown in Figure 3 is the effect of shear rate on the viscosity of the STF samples. It was observed that at 30% STF or 30% w/w concentration of nanosilica in PEG that there was minimal deviation from Newtonian fluid behavior, as the maximum change in viscosity was measured at 0.0475 Pa.s. However, as the concentration of nanosilica increased, the non-Newtonian behavior was much more evident. The shear thickening significantly affected the viscosity change observed in the experiment as well as the shear rate required to induce shear thickening. It was seen that for higher concentrations the samples initially showed shear thinning behavior before reaching a critical shear rate, at which thickening or an increase in viscosity was observed. Thus, for 35% STF, 40% STF, 45% STF, and 50% STF the maximum change in viscosity were measured to be 0.3269 Pa.s, 0.7831 Pa.s, 8.938 Pa.s, and 59.787 Pa.s, respectively. The approximate shear rate required to induce a shear thickening effect was found to be 8.5 s<sup>-1</sup>, 6.1 s<sup>-1</sup>, 2.0 s<sup>-1</sup>, and 1.5 s<sup>-1</sup> for 35% STF, 40% STF, 45% STF, and 50% STF, respectively.



Figure 3. Shear rate effect on viscosity at 25 °C.

Figure 4 shows the viscosity measurements obtained as a function of shear stress. Viscosity was seen to react in a very similar manner to shear stress as it did to shear rate.





Figure 5 shows the effect of shear rate on the viscosity of the STF samples at an elevated temperature of 50 °C. The trends for each sample were similar to what was observed at 25 °C with some changes in critical shear rate and the extent the STF will increase in viscosity under the tested conditions. The maximum changes in viscosity were found to be 0.0558 Pa.s, 0.1001 Pa.s, 0.2367 Pa.s, 1.9637 Pa.s, and 27.723 Pa.s in 30%STF, 35%STF, 40%STF, 45%STF, and 50%STF, respectively. The approximate critical shear rates were found to be 8.9 s<sup>-1</sup>, 8.2 s<sup>-1</sup>, 4.2 s<sup>-1</sup>, and 3.1 s<sup>-1</sup>. These values correspond to a 4.7%, 34.4%, 110.0%, and 106.7% increase in the shear rate required to induce shear thickening in 35%STF, 40%STF, 45%STF, and 50%STF, respectively, with an increase of 25 °C. These results indicate that temperature can have a significant effect on the rheological properties of STFs.



Figure 5. Effect of shear rate on viscosity at 50 °C.

Figure 6 shows the measured viscosity as a function of shear stress. Again, it was seen that viscosity reacted very similarly to shear stress as did shear rate at an elevated temperature.



Figure 6. Effect of shear stress on viscosity at 50 °C.

## 3.2. Puncture Testing

Puncture testing was performed to evaluate the materials' resistance to stabbing on five specimens per sample. The resultant force and displacement from the puncture testing of flax and STF-treated flax data is summarized in Table 1. It was observed that the impregnation of STF into the flax fabric significantly increased the force to puncture the materials. As the concentration of the STF suspension increased from 30% wt/wt nanosilica in PEG to 70% concentration, the impregnated fabrics' puncture resistance increased as well. Puncture resistance for 30% STF flax, 50% STF flax, and 70% STF flax increased 144.1%, 182.4%, and 254.6%, respectively, compared to neat flax. This is similar to reports in the literature for Kevlar<sup>®</sup> fabrics impregnated with STF. A study by Kalman et.al showed a 224.4% increase in peak force to puncture Kevlar® fabric when impregnated with a 31.5wt% SiO<sub>2</sub>/PEG based STF [9]. These values indicate that impregnation of STF into flax fabric is extremely effective in increasing the material's puncture resistance, making flax fabrics impregnated with STF an effective material for use against stabbing threats. The amount of displacement the material can undergo before puncture is also increased with STF impregnation. For neat flax fabric the average fabric displacement at maximum force applied before puncture was measured at 10.15 mm. For 30% STF flax, 50% STF flax, and 70% STF flax this value was measured at 12.18 mm, 13.37 mm, and 13.34 mm, respectively. These values correlate to a 20.0%, 31.7%, and a 31.4% increase measured in the fabric displacement at maximum force compared to neat flax. These results show that the material was still flexible after impregnation with STF. This has also been observed by Kalman et al., and when a 31.5wt% STF suspension was used in Kevlar<sup>®</sup> fabric the displacement at peak force was increased 25.76% [9].

Table 1. Summarized Results of Flax and STF Impregnated Flax Fabric Puncture Testing.

	Average Maximum Force (N)	Standard Deviation	Average Displacement at Maximum Force (mm)	Standard Deviation
Neat flax	279.30	45.90	10.15	1.10
30% STF flax	681.80	69.78	12.18	1.67
50% STF flax	788.85	43.30	13.37	0.55
70% STF flax	990.51	142.67	13.34	0.09

The force displacement data for Kevlar<sup>®</sup>, flax/Kevlar<sup>®</sup> hybrid fabrics, and STFimpregnated hybrid fabrics are summarized in Table 2. Again, the impregnation of STF into the fabrics significantly increased the force to puncture the fabric materials. As the concentration of the STF used to impregnate the fabrics increased, the force to puncture increased as well. For the neat Kevlar® sample, the average maximum force required to puncture the samples was measured at 404.5 N. The average maximum force required to puncture the neat hybrid sample was measured at 232.37 N which, oddly, is lower than the average maximum force of 279.30 N required to puncture neat flax fabric. When the hybrid material was impregnated with STF, large increases in puncture resistances were obtained. For the 30% STF hybrid sample, the average maximum force required to puncture the material was measured at 1641.62 N. This corresponded to a 606.5% increase in the force required to puncture the material compared to the neat hybrid, a 305.8% increase over neat Kevlar®, and a 140.8% increase over the 30% STF flax sample. For the 50% STF hybrid sample, the average maximum force required to puncture the material was measured at 1929.93 N. This corresponds to a 730.5% increase over the neat hybrid, a 491.0% increase compared to neat Kevlar®, and a 144.7% increase compared to 50% STF flax. For the 70% STF hybrid sample, the average maximum force required to puncture the sample was measured at 2210.36 N. This corresponded to an increase of 851.2% over the neat hybrid sample, a 446.4% increase over neat Kevlar<sup>®</sup>, and a 123.2% increase compared to the 70% STF flax sample.

Table 2. Summarized Results of Kevlar® and Hybrid STF Sample Puncture Resistance Results.

	Average Maximum Force (N)	Standard Deviation	Average Displacement at Maximum Force (mm)	Standard Deviation
Neat Kevlar <sup>®</sup>	404.50	67.50	11.60	0.52
Neat hybrid	232.37	69.52	9.71	1.99
30% STF hybrid	1641.62	85.01	15.51	0.89
50% STF hybrid	1929.93	161.41	14.48	0.25
70% STF hybrid	2210.36	307.82	13.13	0.52

An increase in fabric displacement at the average maximum force to puncture was also observed in the hybrid samples. The average displacements at the maximum force required for puncture were found to be 9.71 mm and 11.60 mm for the neat hybrid and Kevlar<sup>®</sup> samples, respectively. The average displacements found at the maximum force required for puncture were found to be 15.51 mm, 14.48 mm, and 13.13 mm for the 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid samples, respectively. These values correspond to an increase in displacement at puncture strength compared to the neat hybrid of 59.7%, 49.1% and 35.2% for the 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid, and 70% STF hybrid samples, respectively. When compared to the neat Kevlar<sup>®</sup> fabric, the increases were found to be 33.7%, 24.8%, and 13.2% for the 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid samples, respectively. These results showed with the impregnation of STF the fabric materials could undergo a greater displacement before being punctured; thus, the materials still would remain flexible enough for use in soft body armor applications.

Results displayed the effectiveness of hybridization of the fabrics when impregnated with STF, as the hybrid samples showed much larger increases in puncture resistance compared to the unhybridized flax samples relative to the neat fabric samples. The effectiveness of impregnating the STF on the puncture resistance was improved with hybridization of the fabric materials as the hybrid samples showed much higher force to puncture than the STF-impregnated flax samples. Hybridized fabric materials impregnated with STF show much promise for use in puncture resistant applications. It is hypothesized that hybridizing STF-impregnated Kevlar<sup>®</sup> fabric with STF-impregnated flax fabric significantly improved puncture resistance due Kevlar<sup>®</sup> being a fabric material with better mechanical properties than flax and STF enhancing the Kevlar<sup>®</sup> fabric more significantly than the flax fabric.

When evaluating puncture resistance of the materials tested, the specific properties were to be reported as well. This specific puncture strength is a more accurate representation of the effectiveness of the STF impregnation. Although large increases in puncture resistance were measured, the increased weight of the STF-impregnated samples needed to be considered as well. The specific puncture strength of the materials was calculated by dividing the force required to puncture the material by the material's areal density. The equation used to calculate specific puncture strength is shown below in Equation (1), with F being the average maximum force required to puncture the material in Newtons and  $\rho$  being the areal density of the material in kg/m<sup>2</sup>.

Specific Puncture Strength = 
$$F/\rho$$
, (1)

Figure 7 shows a comparison of the specific puncture strength of the materials. Significant increases in specific puncture strength were seen with the impregnation of STF into the fabric materials. As the concentration of the STF used to impregnate the fabrics increased, the specific puncture strength increased as well. The specific puncture strength of the neat flax fabric sample was calculated to be 677.92 N/kg/m<sup>2</sup>. The specific puncture strength of the 30% STF flax, 50% STF flax, and 70% STF flax samples were calculated at 828.43 N/kg/m<sup>2</sup>, 1016.56 N/kg/m<sup>2</sup>, and 1151.75 N/kg/m<sup>2</sup>, respectively. These values corresponded to an increase in specific puncture strength compared to neat flax of 22.2%, 50.0%, and 69.9% for 30% STF flax, 50% STF flax, and 70% STF flax, and 70% STF flax, respectively. The increase in specific puncture strength was significantly less than the base increase in puncture strength.



Figure 7. Comparison of specific puncture strengths.

The specific puncture strength of the neat hybrid sample was calculated at 533.58 N/kg/m<sup>2</sup>. The specific puncture strengths of the 30% STF hybrid, 50% STF hybrid and 70% STF hybrid samples were calculated to be 2264.31 N/kg/m<sup>2</sup>, 2807.17 N/kg/m<sup>2</sup>, and 2866.88 N/kg/m<sup>2</sup>, respectively. Compared to the neat hybrid, this corresponded to an increase in specific puncture strength of 324.4%, 426.1%, and 437.3% for the 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid samples, respectively. The specific puncture strength of the Kevlar<sup>®</sup> sample was calculated to be 881.26 N/kg/m<sup>2</sup>. When compared to the neat Kevlar<sup>®</sup> fabric the percentage increases in specific puncture strength for the 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid were found to be 156.9%, 218.5%, and 225.3%, respectively. Again, the increase in specific puncture strength was found to be much less than the increase in puncture strength for the hybrid samples.

Figure 8 shows the average energy absorption of the samples during puncture testing. It was found that as the concentration of the STF suspension increased, the energy absorp-



tion of the impregnated samples also increased. Flax samples impregnated with STF were able to absorb significantly more energy than neat Kevlar<sup>®</sup> specimens.

Figure 8. Comparison of average energy absorbed during puncture testing.

#### 3.3. Ballistic Impact Testing

High velocity ballistic impact testing was performed on three specimens per sample. A projectile delivered with 107.406 J of kinetic energy was stopped with four layers of neat flax. The 30% STF flax, 50% STF flax, and 70% STF flax samples were able to stop projectiles delivered with 121.2 J, 184.680 J, and 240.969 J of kinetic energy, respectively. These values corresponded to an increase in ballistic impact energy absorption of 12.8%, 71.9%, and 124.3% for 30% STF flax, 50% STF flax, and 70% STF flax samples, respectively. Figure 9 shows a graphical comparison of the results. These results prove that impregnating flax fabric with STF significantly improved their ballistic impact resistance.



Figure 9. Comparison of ballistic impact energy absorption for flax fabric samples.

Ballistic impact testing was also performed on hybrid samples. The neat hybrid sample was able to stop a projectile that was delivered with 482.77 J of kinetic energy. The 30% STF

hybrid, 50% STF hybrid, and 70% STF hybrid samples were able to stop projectiles delivered with 602.78 J, 961.04 J, and 1402.48 J of kinetic energy, respectively. This corresponded to an increase in ballistic impact energy absorption of 24.9%, 99.1%, and 190.5% for 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid samples, respectively. Figure 10 shows a graphical comparison of the results. These results show that hybridization of flax with Kevlar<sup>®</sup> significantly improved the ballistic impact resistance of the material.



Figure 10. Comparison of ballistic impact energy absorption for hybrid samples.

As impregnating fabrics with STF increases the weight of the material, the ballistic impact testing evaluated how the increased weight affects the performance of the materials. The specific energy absorption was calculated using Equation (2), where E was the impact energy absorbed in Joules and  $\rho$  was the areal density in kg/m<sup>2</sup>.

Specific Puncture Strength = 
$$E/\rho$$
, (2)

It was found that neat flax had a specific ballistic impact energy absorption per layer of 260.69 J/kg/m<sup>2</sup>. The 30% STF flax, 50% STF flax, and 70% STF flax samples' specific impact energy absorptions per layer were calculated to be 147.27 J/kg/m<sup>2</sup>, 237.99 J/kg/m<sup>2</sup>, and 280.18 J/kg/m<sup>2</sup>, respectively. These results showed that although STF-treated flax absorbed more energy, it did so at the expense of increased weight. Only the 70% STF flax sample was shown to be more effective at absorbing energy from ballistic impact than the neat flax sample on a per weight basis. Even then, it was only an increase of 7.5%. This means that impregnation of STF into flax fabrics did not have significant improvement in the specific ballistic impact energy absorption of the material.

The neat hybrid sample's specific impact energy absorption per layer was calculated to be 1108.53 J/kg/m<sup>2</sup>. The 30% STF hybrid, 50% STF hybrid, and 70% STF hybrid were found to have specific impact energy absorption per layer values of 831.42 J/kg/m<sup>2</sup>, 1397.87 J/kg/m<sup>2</sup>, and 1819.04 J/kg/m<sup>2</sup>. The 30% STF hybrid was not as effective in terms of impact energy absorption per unit weight; however, the 50% STF hybrid and 70% STF hybrid showed significant improvement. The 50% STF hybrid and 70% STF hybrid showed increases in specific ballistic impact energy absorption per layer of 26.1% and 64.1%, respectively. These results showed that the hybridization of flax with Kevlar<sup>®</sup> fabrics impregnated with STF improved the materials' ballistic impact resistance if the concentration of STF used was high enough. The results also displayed the hybrid STF-impregnated materials' promise for use in soft body armor applications. Figure 11 shows a comparison of all samples' specific impact energy absorption per layer.



Figure 11. Comparison of specific ballistic impact energy absorption per layer.

#### 3.4. SEM Imaging

Scanning electron microscope images were taken of the STF-impregnated flax fabric samples. These can be seen below in Figure 12. As is common when working with nanoparticles, agglomerations of the nanosilica particles were seen throughout the samples, with larger agglomerates seen at the higher STF concentrations. The nanosilica/PEG STF suspension was viewed relatively well dispersed throughout the fabric.



**Figure 12.** SEM images of (**a**) nanosilica particle agglomerates, (**b**) 30% STF flax, (**c**) 50% STF flax, (**d**) 70% STF flax.

Figure 13 shows the nanoparticle adherence to individual fibers in the fabric materials. At all STF concentrations it was seen that the fibers were never completely coated in nanoparticles. It was observed that at concentrations of 30% and 50% nanosilica STF the nanoparticle agglomerates and fiber surface coverage were similar. At 70% nanosilica concentration the fiber surface was more completely covered with more agglomerates.



Figure 13. Nanosilica adherence to individual fibers. (a) 30% STF flax, (b) 50% STF flax, (c) 70% STF flax.

The higher coverage of nanoparticles may have been a larger influence on performance than the STF materials they were dispersed in. With a higher dispersion of particles, the inter-yarn friction within the fabric may have reduced the ability of the individual fibers to move as they were more covered in particles. Additionally, the particles would have had the opportunity to embed themselves into the fibers with the application of a force, thus increasing the friction required to move the fibers within the fabric. Because of the higher resiliency of Kevlar<sup>®</sup> fibers, they may have been more receptive to the force of embedding particles vs. flax fibers, which would have been more easily crushed.

#### 4. Conclusions and Future Recommendations

STF-impregnated flax fabrics exhibited significant increases in puncture resistance. As the concentration of the STF used to impregnate the flax fabrics was increased, the puncture resistance was also increased. Although the addition of STF to the flax fabrics adds a significant amount of weight to the sample, the STF-treated flax fabrics still showed significant increases in specific puncture strength. STF-treated flax fabrics also showed an increase in ballistic energy absorption, although with the significant increase in weight from the STF, the treated flax samples did not exceed the performance of the neat flax in terms of specific impact energy absorption.

STF-impregnated hybrid samples of flax/Kevlar<sup>®</sup> fabrics also showed significant increases in puncture resistance far beyond those of just the flax treated with STF. The hybridization of Kevlar<sup>®</sup> with flax fabrics significantly improves the flax fabric's puncture resistance and also its specific puncture strength of the flax fabric. The STF-treated hybrid fabrics also absorbed significantly more energy than untreated flax, Kevlar<sup>®</sup>, and hybrid samples. The specific impact energy absorption per layer in the samples treated with 50% and 70% STF was also measured to be significantly greater than any untreated samples.

The results from this novel study show that treating flax fabrics with STFs does indeed affect their puncture resistance and ballistic impact resistance. The hybridization of Kevlar<sup>®</sup> with flax fabric and treatment with STF leads to significant increases in the properties of the material allowing it to outperform neat Kevlar<sup>®</sup> fabric. This hybrid material could be a candidate for further study in body armor applications, as the material can be easily encased inside a tactical vest or, if needed, vacuum-sealed inside a plastic film and then inserted in tactical vest pouches.

It is the opinion of the authors that the greatest improvement of puncture and ballistic impact properties of fabric type materials is not necessarily the incorporation of a shear thickening fluid into the fabric, but dispersion of particles inside the fabric material. The dispersion of particles increases the puncture resistance and impact resistance by increasing the inter-yarn friction within the fabric, or severely reducing any individual fiber's ability to move when particles are dispersed and locking them in place. More irregularly shaped particles dispersed in a fabric could lead to enhanced properties relative to more regularly shaped particles such as nanosilica spheres, provided the particles have a similar or greater hardness than nanosilica. It is also possible that the particles were able to embed themselves into the fibers when force was applied to the fabrics, helping to increase the friction force required to move fibers in the fabric. It is hypothesized that the effect the particles have on the increase in puncture and ballistic resistance is greater in hybrid impregnated samples because the particles have a greater reinforcing effect in Kevlar<sup>®</sup> fabric. This may be because the Kevlar<sup>®</sup> fibers are more resilient and particles are able to embed into them more readily as compared to flax fibers, which are more easily crushed. The synthetic Kevlar<sup>®</sup> fabric also has a much tighter and more consistent weave in the fabric which may lead to particles being able to embed into multiple fibers more easily.

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### References

- 1. Boersma, W.H.; Laven, J.; Stein, H.N. Shear thickening (dilatancy) in concentrated dispersions. *AIChE J.* **1990**, *36*, 321–332. [CrossRef]
- 2. Lee, Y.S.; Wagner, N.J. Dynamic properties of shear thickening colloidal suspensions. Rheol. Acta 2003, 42, 199–208. [CrossRef]
- 3. Seshimo, K. Viscoelastic Damper. U.S. Patent 4,759,428, 26 July 1988.
- 4. Williams, T.; Day, J.; Pickard, S. Surgical and Medical Garments and Materials Incorporating Shear Thickening Fluids. U.S. Patent 2,007,000,440,086, 15 October 2009.
- 5. Gurgen, S.; Kushan, M.C. High performance fabrics in body protective systems. Mater. Sci. Forum. 2016, 880, 132–135. [CrossRef]
- 6. Hassan, T.A.; Rangari, V.K.; Jeelani, S. Synthesis, processing and characterization of shear thickening fluid (STF) impregnated fabric composites. *Mater. Sci. Eng. A* 2010, 527, 2892–2899. [CrossRef]
- Wetzel, E.D.; Lee, Y.S.; Egres, R.G.; Kirkwood, K.M.; Kirkwood, J.E.; Wagner, N. The Effect of Rheological Parameters on the Ballistic Properties of Shear Thickening Fluid (STF)- Kevlar Composites. *AIP Conf. Proc.* 2004, *712*, 288–293.
- Rosen, B.A.; Laufer, C.N.; Kalman, D.P.; Wetzel, E.D.; Wagner, N.J. Multi-Threat performance of Kaolin-based shear thickening fluid (STF)-treated fabrics. In Proceedings of the SAMPE 2007, Baltimore, MD, USA, 3–7 June 2007.
- 9. Kalman, D.P.; Merrill, R.L.; Wagner, N.J.; Wetzel, E.D. Effect of Particle Hardness on the Penetration behavior of fabrics intercalated with dry particles and concentrated particle-fluid suspensions. *Appl. Mater. Interfaces* **2009**, *1*, 2602–2612. [CrossRef]
- 10. Barnes, H.A. Shear thickening ('Dilatency') in suspensions of nonaggregating solid particles dispersed in Newtonian liquids. *J. Rheology* **1999**, *33*, 329–366. [CrossRef]
- 11. Srivastava, A.; Majumdar, A.; Butola, B.S. Improving the impact resistance of textile structures by using shear thickening fluids: A review. *Crit. Rev. Solid State Mater. Sci.* 2012, 37, 115–129. [CrossRef]
- 12. Liu, X.-Q.; Bao, R.-Y.; Wu, X.-J.; Yang, W.; Xie, B.-H.; Yang, M.-B. Temperature induced gelation transition of a fumed silica/PEG shear thickening fluid. *RSC Adv.* 2015, *5*, 18367–18374. [CrossRef]
- 13. Mahfuz, H.; Clements, F.; Stewart, J. Development of Stab Resistant Body Armor using Fumed SiO2 Nanoparticles Dispersed into Polyethylene Glycol (PEG) through Sonic Cavitation. *NSTI-Nanotech.* **2006**, *1*, 358–361.
- 14. Rangari, V.K.; Hassan, T.A.; Mahfuz, H.; Jeelani, S. Synthesis of Shear Thickening Fluid Using Sonochemical Method. *NSTI-Nanotech.* **2006**, *2*, 637–640.
- Afeshejani, S.H.A.; Sabet, S.A.R.; Zeynali, M.E.; Atai, M. Energy Absorption in a Shear-Thickening Fluid. J. Mater. Eng. Perform. 2014, 23, 4289–4297. [CrossRef]
- 16. Park, Y.; Kim, Y.; Baluch, A.H.; Kim, C.-G. Empirical study of the high velocity impact energy absorption characteristics of shear thickening fluid (STF) impregnated Kevlar fabric. *Int. J. Impact Eng.* **2014**, *72*, 67–74. [CrossRef]

- 17. Park, Y.; Baluch, A.H.; Kim, Y.; Kim, C.-G. High Velocity Impact Characteristics of Shear Thickening Fluid Impregnated Kevlar Fabric. *Int. J. Aeronaut. Space Sci.* 2013, 14, 140–145. [CrossRef]
- 18. Khodadadi, A.; Liaghat, G.; Vahid, S.; Sabet, A.; Hadavinia, H. Ballistic performance of Kevlar fabric impregnated with nanosilica/PEG shear thickening fluid. *Compos. Part B Eng.* **2019**, *162*, 643–652. [CrossRef]
- Yeh, S.K.; Lin, J.J.; Zhuang, H.Y.; Chen, Y.C.; Chang, H.C.; Zheng, J.Y.; Yang, L.Y.; Lee, K.C.; Chen, Y.L.; Rwei, S.P. Light shear thickening fluid (STF)/Kevlar composites with improved ballistic impact strength. J. Polym. Res. 2019, 26, 155–167. [CrossRef]
- Lee, Y.S.; Wetzel, E.D.; Wagner, N.J. The ballistic impact characteristics of Kevlar woven fabrics impregnated with a colloidal shear thickening fluid. J. Mater. Sci. 2003, 38, 2825–2833. [CrossRef]
- 21. Majumdar, A.; Butola, B.S.; Srivastava, A. An anlysis of deformation and energy absorption modes of shear thickening fluid treated Kevlar fabrics as soft body armour materials. *Mater. Des.* **2013**, *51*, 148–153. [CrossRef]
- Egres, R.G., Jr.; Decker, M.J.; Halbach, C.J.; Lee, Y.S.; Kirkwood, J.E.; Kirkwood, K.M.; Wagner, N.J.; Wetzel, E.D. Stab Resistance of Shear Thickening Fluid (STF)-Kevlar Composites for Body Armor Applications. *Transform. Sci. Technol. Curr. Future Force* 2006, 264–271. [CrossRef]
- Rao, H.; Hosur, M.V.; Mayo, J.; Burton, S.; Jeelani, S. Stab Characterization of Hybrid Ballistic Fabrics. In Proceedings of the SEM Annual Conference, Albuquerque, NM, USA, 1–4 June 2009; pp. 264–271.
- 24. Kang, T.J.; Hong, K.H.; Yoo, M.R. Preparation and properties of fumed silica/Kevlar composite fabrics for application of stab resistant material. *Fibers Polym.* **2010**, *11*, 719–724. [CrossRef]
- Feng, X.; Li, S.; Wang, Y.; Wang, Y.; Liu, J. Effects of different silica particles on quasi-static stab resistant properties of fabrics impregnated with shear thickening fluids. *Mater. Des.* 2014, 64, 456–461. [CrossRef]
- Qin, J.; Zhang, G.; Zhou, L.; Li, J.; Shi, X. Dynamic/quasi-static stab-resistance and mechanical properties of soft body armour composites constructed from Kevlar fabrics and shear thickening fluids. *RSC Adv.* 2017, 7, 39803–39813. [CrossRef]
- 27. Haro, E.E.; Szpunar, J.A.; Odeshi, A.G. Ballistic impact response of laminated hybrid materials made of 5086-H32 aluminum alloy, epoxy, and Kevlar<sup>®</sup> fabrics impregnated with shear thickening fluid. *Compos. Part A Appl. Sci. Manuf.* **2016**, *87*, 54–65. [CrossRef]
- 28. Gassan, J. A study of fibre and interface parameters affecting the fatigue behavior of natural fibre composites. *Compos. Par A Appl. Sci. Manuf.* 2002, 33, 369–374. [CrossRef]
- 29. ASTM F1342/F1342M-05R22; Standard Test Method for Protective Clothing Material Resistance to Puncture. ASTM: West Conshohocken, PA, USA, 2022. [CrossRef]

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