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Production of Kenaf Fibre Reinforced Polyethylene Composite for Ballistic Protection

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Abstract— Kenaf reinforced polyethylene composites for ballistic protection was produced in this work. The samples of alkaline treated and silane-coupled non-woven matted kenaf fibers were cut to the required dimensions and oriented vertically and horizontally in combinations with a virgin high density polyethylene (VHDPE). The composite panels were produced using Box-Behnken 3-variables settings. The following factors settings were used (molding temperature: 160^0C to 200^0C; molding time: 60minutes to 80minutes; fiber volume fraction: 10% to 30%) based on dry mass in a two-piece mild steel compression molding set. The pressure for heating and cooling was controlled at 12MPa. The responses as tensile and flexural values were determined and optimized. The composite sample of VHDPE ballistic test were blended based on the optimum settings of temperature: 200^0C; molding time: 80minutes and fiber volume fraction:30%) and tested with Jojeff Magnum riffle gun. The mechanical and ballistic properties of the composite panel of VHDPE were determined. The studies revealed that fiber volume at 30% protected against Armor Level Protection Class of NIJ standard level III-A for VHDPE composite.

Keywords—Ballistic Protection, Design of Experiment, Fiber treatments, Kenaf fibers, Polyethylene, Stress properties

1 Introduction

FIBERS were used to reinforce brittle materials in ancient times before the birth of Christ when straws were used to reinforce sun-baked bricks. The last decades had been with an increasing interest in vegetable fibers as replacement materials for synthetic fiber reinforced products. The ancient use of vegetable fibers, which include the making of car mats, rugs, coir mats, floor coverings and furniture, as practiced in Latin America, South East Asia, East Africa etc., in the last decades had been with an increasing interest in vegetable fibers as reinforcing materials for reinforced products.

When natural fibers are closely compared to inorganic fibers, it presents some well-known advantages such as lower density and cost; are less abrasive to the processing equipment, harmless, biodegradable, renewable, and their mechanical properties can be comparable to those of inorganic fibers, furthermore, they are recyclable, easily available in most countries, easy fiber surface modification, and its relative nonabrasiveness [1], [2]. Much work has been done on virgin thermoplastic and natural fiber composites, with successful prove of their application to various fields of technical applicability, especially for load-bearing application [3]. Thermoplastics such as polyethylene (PE) [4], [5], have been compounded with natural fibers (such as wood, kenaf, flax, hemp, cotton, kraft pulp, coconut husk, areca fruit, pineapple leaf, oil palm, sisal, jute, henequen leaf, ovine leather, banana, abaca, and straw) to prepare composites [3].

In recent years advances in material science have opened the door to the old idea of a literal "bulletproof vest" that will be able to stop handgun and rifle bullets with a soft textile vest without the assistance of heavy and cumbersome extra metal or ceramic plating [6]. Bulletproof or "bullet resistant" vests are modern light armor specifically designed to protect the wearer's vital organs from injury caused by firearm projectiles.

This study tends to focus on kenaf (*Hibiscus cannabinus*) in combination with high density virgin polyethylene with a view to determine its mechanical and ballistic properties and expose other areas of further studies. It is well known that the performance of composite depends on the properties of the individual components and their interfacial compatibility. Kenaf bast fiber has been reported to have superior flexural strength combined with its excellent tensile strength that make it the material of choice for a wide range of extruded, molded and non-woven products as widely discussed by other authors. Kenaf reinforced polyethylene composites were produced in a design of experiment using Box-Behnken response surface methodology at factors settings of temperature, molding time and volume fraction.

2 Materials and Methods

2.1 Materials

The materials used are Kenaf fibers, virgin high density polyethylene, VHDPE pellets, from Ariken Ltd (Onitsha), universal mold releaser wax, aluminum foil, sodium hydroxide, acetic acid, silane and ethanol.

2.2 Pretreatment of the fiber

The kenaf fibers grown in the Nsukka Area of Enugu State, Nigeria was provided by Center for Composite Research and Development (CCRD), Enugu State, were soaked in detergent for 30min, washed and thoroughly rinsed with clean water. The fibers were sun dried to remove the moisture content. The removal of moisture content helps to reduce the water absorbed in the cellulose, hemicelluloses and lignin components of the fiber.

2.3 Treatment with sodium hydroxide (NaOH) or mercerization

Kernaf fibers were soaked into NaOH solutions of 1mol for 2hours. After that the fibers were washed with very dilute (1mol) acetic acid to remove the nonreacted alkali and rinsed thoroughly with distilled water. The washed fibers were then sun dried. Mercerization is alkali treatment of natural fibers which leads to fibrillation and causes the breaking down of the composite fiber bundle into smaller fibers. Mercerization

reduces fiber diameter, thereby increases the aspect ratio which leads to the development of a rough surface topography that results in better fiber-matrix interface adhesion and an increase in mechanical properties when compared to other treatment methods as reported by Joseph et al [7]. Moreover, mercerization increases the number of possible reactive sites and allows better fiber wetting.

2.4 Silane coupling

The pretreated fibers were placed into 2% silane solution in water and ethanol (40/60 by volume) mixture for one hour; washed with distilled water, sun dried and wrapped in polythene bag to prevent absorption of moisture from the atmosphere. Different volume ratios (20/80; 30/70; 50/50; 60/40; 70/30; 80/20) were applied in the preliminary stage of this work to determine the volume ratio that would provide the optimum value/best result but 40/60 volume ratio gave the best mechanical property. Coupling agents usually improve the degree of cross-linking in the interface region and offer a perfect bonding. Therefore, the hydrocarbon chains provided by the application of silane restrained the swelling of the fiber by creating a cross-linked network because of covalent bonding between the matrix and the fiber. This corroborated well with the work of Agarwal et al [8] in this area that the hydrocarbon chains provided by the silane application influenced the wet-ability of the fibers, thus improving the chemical affinity to polyethylene.

2.5 Design of Experiment

Samples of alkaline treated and silane-coupled kenaf fibers were reinforced with virgin high density polyethylene in a three-level experimental designs using Box-Behnken 3-variables settings (molding temperature:160^{0}C to 200^{0}C; molding time: 80minutes to 60minutes; fibre volume fraction: 10% to 30%) based on dry mass in a two-piece mild steel molding set. Compression-molding was done in a locally manufactured hot press. The pressure for heating and cooling was controlled at 120 bar (12MPa). The composite sample of VHDPE for ballistic test was blended based on the optimum settings of temperature: 200^{0}C, molding time: 80minutes and fiber volume fraction: 30%. Table 1 showed the levels of variables chosen for the Box–Behnken response surface design.

Table 1

The initial level settings of design properties

Factor	Name	Units	Symbol	Coded variable level		
				Low level	Centre level	High level
A	Temperature	^{0}C	A	160	180	200
B	Time	min	B	60	70	80
C	Volume Fraction	%	C	10	20	30

2.6 Determination of density

The density of a material is the mass per unit volume measured in g/cm^3. The theoretical density of composite materials in terms of weight fraction was obtained as equation (1) given by Agarwal and Broutman [9]:

$$\rho_{ct} = 1/[(W_f/\rho_f) + (W_m/\rho_m)] \quad (1)$$

Where, W and ρ represent the weight fraction and density respectively. The suffix f, m and c stand for the fiber, matrix and the composite materials respectively.

2.7 Determination of tensile strength (ASTM D638)

The testing was done in standard laboratory atmosphere of 23°C ± 2°C (73.4°F ± 3.6°F) and 50 ± 5 percent relative humidity. This condition of plastic for not less than 40 hours prior to test is in accordance with Procedure A of ASTM D638. Monsanto Tensometer Tensile Tester was used at cross-head speed of 50mm/min. The two ends of the test piece were inserted into the jaw of the Monsanto Tensile Tester at cross-head speed of 50mm/min. The specimens were positioned horizontally in the grips of the testing machine. The grips were then tightened evenly and firmly to prevent any slippage with gauge length kept at 50mm.

Specimens with nominal dimensions of 160 mm x 20mm x 3.2mm were used. The basic relationship to determine tensile strength (σ) is Force (load), F per Cross sectional area, A.

$$\sigma = F/A \quad (2)$$

2.8 Determination of flexural strength (ASTM D790)

Flexural test was carried out using the same Monsanto Tensometer Tensile Tester at cross-head speed of 10mm/min. Three-point testing method was used under specimens with nominal dimensions of 300 mm x 20mm x 3.2mm. Each test was repeated three times. Flexural strength and modulus were read off from the computer connected with testing machine. The values were analyzed and plotted. A span of 200mm was taken and cross head speed was maintained at 4mm/min. The strength of a material in bending is expressed as the stress on the outermost fibers of a bent test specimen, at the instant of failure. Equation (3) shows the relationship.

$$\sigma_f = 3FL/2bd^2 \quad (3)$$

Where: σ_f= Flexural stress/strength (MPa), F = Applied load at fracture point to specimen (N), L= Length of support span (mm), b = Width of the test specimen (mm), d = Thickness of the test specimen (mm).

2.9 Ballistic Testing of the Composite Panel (NIJ 0101.06 and 0101.04 Standards)

The ballistic test was carried out on ballistic panel of VHDPE blended based on the optimum settings obtained from the optimization solution. This composite sample with optimum tensile stress was tested with a Jojeff Magnum riffle gun with reference velocity of 440m/s (±15m/s). The panel was fastened on the torso of human doll held on a rigid stand and three series of shots (hits) at different range from muzzle distances starting with shot at "point blank range, PBR of 1.2m (3.9ft)". The penetration depths/levels were measured with micrometer screw gauge and recorded. Testing was based on threat found locally which was in reasonable agreement with

the work of Terry [10] and Nosworth [11].

The transmitted impact energy or back face signature (BFS) was measured by shooting armor mounted in front of a backing material (oil-based modified modeling clay). The clay was used at a control temperature by refrigerating for 12hours before use. After the armor was impacted by the test bullet, the vest was removed from the clay; edge of the deformation was smoothened using a plastic scraper. The depth of deformation/indentation in the clay was measured with a caliper. The US-NIJ standards allow for 44mm. The kinetic energy of the non-penetrating impact was also determined as impact energy.

2.10 Analytical study

The analytical study was done using the Design Expert® software. The model equations were generated in this software. The predicted response surface models were used to predict the tensile strength, flexural strength and optimized response values.

3 RESULTS AND DISCUSSION

Tensile and flexural properties of the composite including the ballistic and impact performance were discussed in this section.

3.1 Modes of failure

The tensile loading causes stresses in all the composites. The results suggest tensile and flexural failures in fibers of the composite as the matrix was observed to play a role in the failure process as load cell increased, resulting in increased matrix damage and bunch fiber pull-out.

3.2 Design of experiment analysis

The response values of the independent variables are depicted in table 2 as tensile strength and flexural strength. The goal of the experiment was to determine the factor settings that would optimize the response variables. The effects were recorded in the response columns.

Table 2
Box-Behnken designed experiment summary

Std	Run	Factor 1 A:Temperature 0C	Factor 2 B:Time Min	Factor 3 C:Volume Fraction %	Response 1 Y1:Tensile MPa	Response 2 Y2:Flexural MPa
1	11	160	60	20	37.34	45.06
2	4	200	60	20	40.28	47.12
3	13	160	80	20	38.13	46.53
4	9	200	80	20	42.28	47.14
5	5	160	70	10	28.63	40.58
6	1	200	70	10	32.31	42.86
7	15	160	70	30	48.06	59.22
8	3	200	70	30	52.16	62.29
9	12	180	60	10	29.53	40.76
10	7	180	80	10	30.69	41.11
11	10	180	60	30	48.63	61.53
12	6	180	80	30	50.81	61.84
13	8	180	70	20	39.16	46.25
14	2	180	70	20	39.16	46.25
15	14	180	70	20	39.18	46.16

The run order indicated the order of data collection.

3.3 Analysis of Variance for Response Y_1 Tensile

Analysis of variance for response Y_1 tensile was shown in Table 3.

Table 3
Analysis of Variance for response Y_1 Tensile

Analysis of variance, ANOVA table for Response Surface Quadratic model

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	805.82	9	89.54	4566.19	< 0.0001	significant
A-Temperature	0.47	1	0.47	24.09	0.0044	
B-Time	0.067	1	0.067	3.40	0.1246	
C-Volume Fraction	0.44	1	0.44	22.31	0.0052	
AB	0.37	1	0.37	18.67	0.0076	
AC	0.044	1	0.044	2.25	0.1940	
BC	0.26	1	0.26	13.26	0.0149	
A^2	0.47	1	0.47	24.12	0.0044	
B^2	1.078E-003	1	1.078E-003	0.055	0.8240	
C^2	2.16	1	2.16	110.32	0.0001	
Residual	0.098	5	0.020			
Lack of Fit	0.098	3	0.033	244.44	0.0041	Significant
Pure Error	2.667E-004	2	1.333E-004			
Cor Total	805.92	14				

The Model F-value of 4566.19 implied the model was significant. There is only a 0.01% chance that an F-value this large could occur due to noise values of "Prob > F" less than 0.0500. This indicated model terms were significant. In this case A, C, AB, BC, A^2, C^2 were significant model terms. Values greater than 0.1000 indicated the model terms were not significant.

Table 4
Regression analysis for Y_1 tensile

Std. Dev.	0.14	R-Squared	0.9999
Mean	39.76	Adj R-Squared	0.9997
C.V. %	0.35	Pred R-Squared	0.9981
PRESS	1.56	Adeq Precision	204.161

In table 5, the R-squared values of 99% indicated that the polynomial was a very good description of the relationship between these three factors. The Predicted R-Squared of 0.9981 was in reasonable agreement with the "Adjusted R-Squared" of 0.9997; that is, the difference is less than 0.2. The R-squared values of approximately 98-99% implied that the polynomial is a very good predictor of the response as also reported by Montgomery, [12]. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 204.161 indicated an adequate signal. This model can be used to navigate the design space.

Coefficient of Variation, CV value of 0.35% indicated a good degree of precision and reliability with which the treatments were compared and was in reasonable agreement with the maximum acceptable CV value of 30%. The PRESS (Predicted residual sum of squares) was 1.56 which measured how the polynomial model fitted each point in the design.

Table 5
Coefficients estimates of Y_1 tensile

Factor	Coefficient Estimate	Df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	39.17	1	0.081	38.96	39.37	
A-Temperature	1.86	1	0.050	1.73	1.99	1.00
B-Time	0.77	1	0.050	0.64	0.89	1.00
C-Volume Fraction	9.81	1	0.050	9.69	9.94	1.00
AB	0.30	1	0.070	0.12	0.48	1.00
AC	0.10	1	0.070	-0.075	0.28	1.00
BC	0.25	1	0.070	0.075	0.43	1.00
A^2	0.36	1	0.073	0.17	0.55	1.01
B^2	-0.017	1	0.073	-0.20	0.17	1.01
C^2	0.77	1	0.073	0.58	0.95	1.01

From the regression coefficients in Table 6, it could be seen that virtually, all terms have a positive effect on the model. Ideal Variable Inflator Factor, VIF is 1.0. VIFs above 10 are cause for alarm, indicating coefficients are poorly estimated due to multicollinearity (correlation between predictors).
Confidence Interval, CI, represented the amount of error that was allowed in the statistical analysis at 95% standard. The CI columns at low and high have reasonably low values for all terms which gave an impression of the precision of the parameters estimated. Final equation in terms of coded factors was given in equation (4):

$$Y1 = +39.17 + 1.86 * A + 0.77 * B + 9.81 * C + 0.30 * AB + 0.10 * AC + 0.25 * BC + 0.36 * A^2 - 0.017 * B^2 + 0.77 * C^2 \quad (4)$$

The equation in terms of coded factors generated was used to make predictions about the response for given levels of each factor. The coded equation was useful for identifying the relative impact of the factors by comparing the factor coefficients.
Final equation in terms of actual factors:

$$Y1 = +53.1825 - 0.3456 * A - 0.2227 * B + 0.4021 * C + 0.0015 * A * B + 0.0005 * A * C + 0.0026 * B * C + 0.0009 * A^2 - 0.0002 * B^2 + 0.0077 * C^2 \quad (5)$$

The equation in terms of actual factors was used to make predictions about the response for given levels of each factor. The values were shown in table 6. Here, the levels were specified in the original units for each factor. This equation was not used to determine the relative impact of each factor because the coefficients were scaled to accommodate the units of each factor and the intercept was not at the center of the design space. The equation is mathematical relationship generated using regression analysis for the studied response variables.

Using response Y_1 (tensile) as optimization objective, the following summary statistics were selected: temperature: 200.000, time: 80.000, Volume fraction: 30.000, Y_1 tensile: 53.373, Y_2 flexural: 61.937

Therefore, the optimal settings were A=200^{0}C, B=80min and C=30%. To confirm this conclusion, an experiment was conducted using these settings and result of the response variables compared favorably with the predicted response variables. The settings were also used to blend the ballistic composite panels. Y_2: Flexural Final Equation in Terms of Coded Factors:

$$Y2 = +46.22 + 1.0025 * A + 0.2688 * B + 9.9463 * C - 0.36 * AB + 0.1975 * AC - 0.0021 * BC + 0.085 * A^2 + 0.16 * B^2 + 4.93 * C^2 \quad (6)$$

The equation (6) in terms of coded factors can be used to make predictions about the response for given levels of each factor. The coded equation was useful for identifying the relative impact of the factors by comparing the factor coefficients.
Final Equation in Terms of Actual Factors:

$$Y2 = +30.3338 + 0.0808 * A + 0.1346 * B - 1.1491 * C - 0.0018 * A * B + 0.001 * A * C - 0.0001 * B * C + 0.0002 * A^2 + 0.0016 * B^2 + 0.0493 * C^2 \quad (7)$$

The equation (7) in terms of actual factors was used to make predictions about the response for given levels of each factor. Here, the levels were specified in the original units for each factor. This equation was not used to determine the relative impact of each factor because the coefficients were scaled to accommodate the units of each factor and the intercept was not at the center of the design space.

Table 6
Predicted response surface model of Y_1 tensile

Std	Run	Factor 1 A:Temperature ^{0}C	Factor 2 B:Time min	Factor 3 C:Volume Fraction %	Response 1 Y1:Tensile MPa	Model/Pred. Response Y1:Tensile MPa
1	11	160	60	20	37.34	37.09
2	4	200	60	20	40.28	40.22
3	13	160	80	20	38.13	38.00
4	9	200	80	20	42.28	42.25
5	5	160	70	10	28.63	28.57
6	1	200	70	10	32.31	32.10
7	15	160	70	30	48.06	48.01
8	3	200	70	30	52.16	52.00
9	12	180	60	10	29.53	29.50
10	7	180	80	10	30.69	30.40
11	10	180	60	30	48.63	48.63
12	6	180	80	30	50.81	50.57
13	8	180	70	20	39.16	39.01
14	2	180	70	20	39.16	39.01
15	14	180	70	20	39.18	39.01

From Table 3.2e, the predicted polynomial model values were close to the actual values which validated the adequacy of the model. The optimum predicted modeled value at factor settings of T=200 ^{0}C, t_m=80minutes and V_f=30% was 53.20MPa which was in reasonable agreement with the result of optimum selected solution of 53.37MPa.

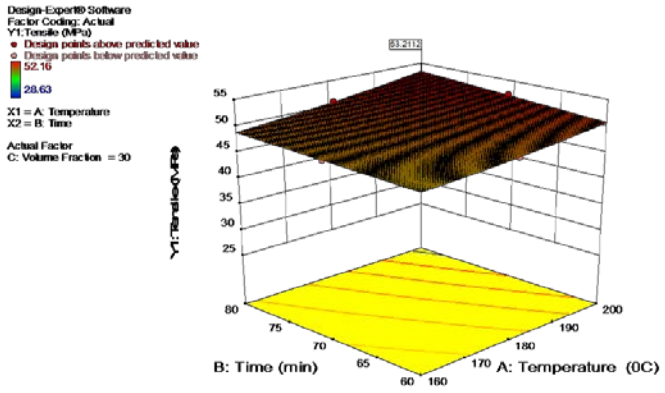

Fig1. Response surface plot showing the influence of two different factors levels on response Y_1 tensile

A 3D-response surface plot in Fig 1 revealed that at the fiber volume of 30% that increased temperature and molding time had positive influence on the tensile stress and more effective at maximizing yield. However, the effect of temperature seemed to be more pronounced as compared with that of time. This also indicated that the settings achieved favorable results for all responses.

The effects of factor levels combination on the response variable were explained by the fact that tensile strength increases with increased temperature due to the facts that press temperature led to decrease in water absorption and thickness swelling; and better adhesion between fibers and matrix. Also polymerization required sufficient time for heat to move through the mat. Increasing the fiber volume to 30% strengthened the internal bonding and improved the mechanical strength and physical properties of the composites. Pre-experimental trial revealed that matrix composite tensile strength increased with the amount of kenaf fiber up to a certain threshold of 30% fiber loading and decreased with further loading which indicated ineffective stress transfer between the fiber and matrix as also reported by Shaikh et al [13][. Thermogravimetric (TG) analysis from the previous works indicated that the kenaf fibers were thermally stable below 218 ^{0}C and that, as such, the fibers could be effectively used as reinforcement when the molding temperature was set under this temperature as stated also in the work of Cao et al [14].

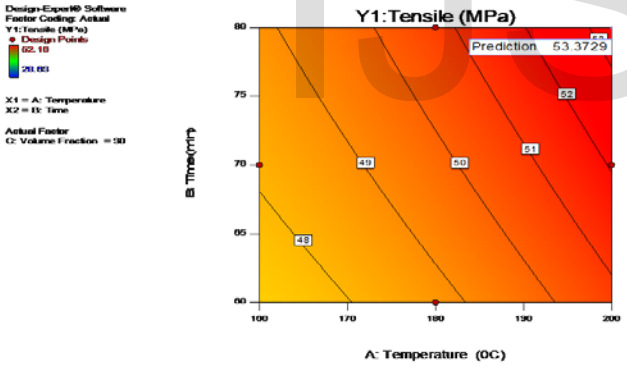

Fig2. Contour plot showing the relationship between various levels of two factors on response Y_1 tensile

The contour plot in Fig2 exhibited the similar trend observed from response surface plot. It clearly confirmed that tensile stress increased towards the optimum predictors or independent variables.

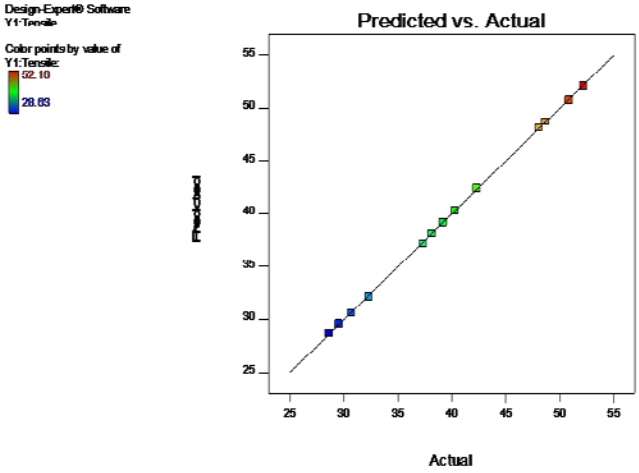

Fig3. Predicted versus Actual plot of Y_1

This diagnostics externally studentized plot of predicted versus actual validated the model and confirmed the adequacy of the model as most of the observed values in Fig 3 clustered closely to the regression line.

3.4 Ballistic Tests

The ballistic panel of VHDPE showed penetration/ballistic resistance with three different hits (shots) in Table 7. This could be explained by the fact that when the bullet struck the body armor, it is caught in a "web" of very strong fibers. These fibers absorb and disperse the impact energy that is transmitted to the ballistic panel from the bullet, causing the bullet to deform or "mushroom." Additional energy is absorbed by each successive layer of material in ballistic vests, until that time as the bullet has been stopped.

Because the fibers work together both in individual layers and with layers of the matrix in the vest, a large area of ballistic vest becomes involved in preventing the bullet from penetrating. This also helps in dissipating the forces which can cause non penetrating injuries (what is commonly referred to as "blunt trauma") to internal organs.

Table 7
VHDPE ballistic tests results

Test No.	Caliber	Test Range		Penetration Depth/Level (mm), VHDPE	Penetration Depth/Level (mm), RHDPE	Penetration Resistance
		Standing Distance (m)	Nozzle Distance (m)			
1	9mm (.357)	2.20	1.20	6.50	6.00	Yes
2	9mm (.357)	3.20	2.20	5.50	5.10	Yes
3	9mm (.357)	4.20	3.20	4.30	4.20	Yes

3.5 Physical properties of VHDPE ballistic composite

The results of physical properties of VHDPE ballistic composite panel are shown in Table 8.

Table 8
Physical properties of VHDPE ballistic composite

Ballistic composite type	Color	Density (g/cm^3)	Dimensions (cm)
VHDPE	White	0.95	16.5cm×16.5cm×2.5cm

3.6 Impact Specimen Test

The transmitted impact energy or back face signature (BFS) for the ballistic composite was low and within the limit of 44mm US-NIJ standards as depicted in Table 9. This indicated blunt trauma protection (from internal injury resulting from the impact energy) by the ballistic panel.

Table 9
Impact Test Results

Composite type	Projectile type	Mass of projectile (g)	Impact velocity (m/s)	Cavity depth (mm)	Impact energy (J)
VHDPE	9mm	8	440	24.05	774.4

The plates shown are untreated kenaf fiber strands, VHDPE ballistic composite panel, VHDPE ballistic composite panel with shots and ballistic vest.

Fig3. Kenaf fibre strands

Fig4. VHDPE Ballistic panel

Fig5. VHDPE panel with shots

Fig6. Ballistic panel vest

4 CONCLUSION

Kenaf fiber (Hibiscus cannabinus), has several characteristics which favor its industrial use, in general, and in particular as a raw material for ballistic vest (armor). Samples of kenaf fibers were characterized and treated as good surface treatment was required to properly combine hydrophilic fiber and hydrophobic polymer to produce composite with excellent properties. The studies of factors levels settings: temperature, time and fiber volume effects revealed that suitable fiber volume at 30% for VHDPE composite protected against Armor Level Protection Class: NIJ standard level III-A. The strength of this composite could be also attributed to high pressure value of 12MPa as more individual fibers were compressed, thus higher percentage of overlap between fibers which resulted in better fiber-to-fiber orientation and arrangement. Kenaf fiber which is underutilized constitutes a greater economic potential and a strong economic driver, at least initially, as a result of its potential in industry. Kenaf fiber can be used in ballistic protection design. The findings from the study would be a spring board to further research works for academic and industrial purposes, especially for students and scholars.

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