

Investigating The Mechanical Properties of Sisal/Areca/Sisal Natural Fiber Reinforced Composite Material Treated with NaOH and Filled with Al₂O₃ and TiO₂

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Abstract

Biopolymer-based composites which are obtained from the natural resources like plants, animal or mineral based have recently attracted significant interest because they can be used in interesting ways in biology and materials science. These materials are generally non-toxic, reusable, and recyclable, which are useful in many situations where sustainability and environmental protection are important. Plant Based fibers like Bamboo, Coir, Kenaf, Banana, Sisal, hemp and jute are proving their potentiality in major industrial, home based and automotive applications. Sisal Fibers are prominently used as the as a replacement for synthetic materials like glass and carbon fiber due to its favorable physical and mechanical properties, environmental friendliness, cost effectiveness and bio-durability. Moreover, addition of fillers enhance the properties of natural fiber composites. The primary objective of the research is to study how NaOH treatment and fillers of Al₂O₃ and TiO₂ affect the mechanical properties of a composite material based on Sisal-Areca fibers. The composites are made using a hand layup technique, where Composition 1 consists of untreated raw fiber (USAS), Composition 2 is fiber treated with NaOH (SAS), Composition 3 contains fiber with Al₂O₃ fillers (SASAL), and Composition 4 has TiO₂ fillers (SASTI). The standards followed for testing include tensile testing (ASTM 638), flexural testing (ASTM 570), impact testing (ASTM 256), density measurement (ASTM 792), and hardness testing (ASTM D2240). The addition of Al₂O₃ and TiO₂ fillers improves the tensile strength of the material by 8.6% and 11.09%, respectively. The fillers of Al₂O₃ improve the flexural strength by 54%, and TiO₂ enhances it by 66.83%. Furthermore, the impact strength is improved by up to 57.2% for Al₂O₃ fillers and 67.28% for the addition of TiO₂ fillers. The addition of fillers enhances the mechanical properties of the material with an increase in density.

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INTRODUCTION

Natural fibers can originate from plants, animals, or minerals. Those with a vegetable origin are composed of cellulose and lignin and can be fibrous or non-fibrous. Natural fibers are

attractive due to their biodegradability, lightweight, low cost, recyclability, safety for the environment and humans, compatibility, hydrophilic nature, low density, commercial availability, high impact resistance, high flexibility, low specific gravity, less abrasiveness to equipment, process-friendly, lower greenhouse emissions, recyclability, and CO₂ neutrality [1-6]. Various natural fibers can be obtained from different parts of plants, as shown in Table 1. Hybrid composites provide superior and combined properties compared to conventional monolithic composites [7]. The reinforcement of more than one type of material in the matrix forms a hybrid composite that is moderately cheap using the different techniques like chemical treatments and coating of different materials on natural fibers which enhance the mechanical properties of natural fibers [8] which are in form of seed, leaves, grass, fruit or bast [9] having water opposing properties of composite materials [10] and also can be considered as a possible alternative to exchange the costly synthetic fibers like carbon in retrofitting applications [10] and use of bio composites represent a green and eco-friendly composites [11]. One of the advantage of Switching to Natural fibers are that they are resistant to fire and if burnt, they do not produce poisonous gases [12].

MATERIALS

The focus of this research is to explore how treating fibers with sodium hydroxide (NaOH), as well as incorporating fillers of Al₂O₃ and TiO₂, impacts the mechanical properties and density of a composite material. The materials utilized in the study include Sisal Fiber, Areca Fiber, Epoxy Resin, Al₂O₃, TiO₂, (shown in Figure 1) and sodium hydroxide. The objective of the research work is to investigate how these variables affect the tensile, flexural, and impact strength of the material. The role of NaOH treatment and fillers of Al₂O₃ and TiO₂ in enhancing the strength of natural fiber-based composites, is studied and the way for developing sustainable and cost-effective materials for various industries.

Table 1. Types of fiber.

No.	Fiber	Example of fiber	References
1.	Seed	Cotton, Kapkok, Coir	[4,11]
2.	Leaves	Pineapple, Banana sisal, abaca, palm, curaua, agaves	[4,5,11]
3.	Grass	wheat, corn, rice, Oat, Barley, Bamboo, Bagasse, Rape, rye, maize	[4,5,9,11]
4.	Fruit	Coir, Luffa	[5]
5.	Bast	Flax, Jute, Hemp, kenaf, cane, ramie, canabis	[3,6,9,11]



Figure 1. a) Raw Sisal, b) Areca, c) Epoxy resin, d) NaOH, e) Al₂O₃, f) TiO₂
Sisal Fiber

Sisal Fiber are widely used as reinforcement in polymer composites due to its strength, less density, friendliness with environment, high Specific-strength, cost effectiveness and sisal fiber has also proved its potential as replacement of synthetic materials like Glass fiber and carbon fiber due to its promising mechanical and physical characteristics like flexural and tensile strength, durability and biodegradability. [13]. Structural sisal fiber is of commercial interest due to its durability and resistance to splitting or fibrillation during the extraction process [14]. The mechanical properties of sisal fiber-reinforced composites can be influenced by various factors such as fiber orientation, length, and alkali treatment [15]. Sisal fiber (Fig.1a) exhibits properties equivalent to those of polypropylene when used in the appropriate fiber proportion [16]. The incorporation of pure sisal fiber with jute and glass fiber results in superior properties [17]. Sisal fiber is less expensive and can be utilized in hybrid composites when combined with human hair, resulting in better properties [18]. Hybridization of sisal with glass fiber enhances the tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength of the composite [19].

Areca Fiber

Areca fibers (Figure 1b) are a type of natural fiber obtained from the shell of the Areca Catechu Linnaeus plant, which is a fibrous material that exhibits tensile strength ranging from 147-322 MPa [20]. However, when reinforced with polypropylene at levels exceeding 5%, the mechanical properties of the composite decrease [21]. NaOH treatment of Areca fibers can enhance the material's properties and increase the adhesiveness of composites [22-23]. Areca fibers are dry in nature, which enables the fabrication of better composites [24]. By using chemical treatment, a significant increase in the impact strength of Areca composites is achieved [25].

Al₂O₃

Al₂O₃ (Figure 1e.) is a versatile material with excellent mechanical and thermal stability, as well as an acid-base surface, making it a commonly used catalyst and absorbent [26]. When hybridized with composite materials using gradually increasing percentages, Al₂O₃ enhances the tensile, flexural, and tribological properties of the resulting material, as demonstrated in multiple studies [27, 28, and 29].

TiO₂

TiO₂ (Figure 1f) has some specific advantages like nontoxicity, chemical stability, corrosion resistant good electrical properties, and compatibility with different materials. TiO₂ as fillers increases

the tensile and flexural properties of the material [30-32].

Epoxy Resin

Epoxy (Figure 1c) is bonding agent which plays a vital role in fabrication of the bio-composite for handlayup manufacturing process in which epoxy resin increases the bondage between the fibers [33]. Epoxy resins are thermosetting resins that are converted into hard cured material under the application of curing methods. They are normally in the liquid form or soft solid. [34].

MANUFACTURING METHODS

Alkali Treatment

The main techniques are alkali treatment – mercerization, acetylation, benzylation, permanganate, silane, peroxide, enzyme, isocyanate, treatment and esterification which promote better adhesion between the matrix, cleaning of moistures, dirt and impurities [35, 36]. In the current experimental work sodium hydroxide (NaOH) to remove the cellulose from the natural fiber. NaOH has pH value as 6.5% to 8.5% after soluble in water, so it can't affect the environment even poured the treated water on the land after the treat. . The Areca fiber and Sisal fiber were dipped in the 10% Solution of NaOH for 3 hours [35, 36] and later on removed and cleaned with fresh water and then kept in the sunlight for 24 hours for drying and removal of moistures. The alkaline treatment of NaOH enhances the properties of fibers. In the experimental process, Powder form of NaOH is purchased from local market and mixed with distilled water in proportion of 1 Liter of water with 200 gram of sodium hydroxide. The mixture was mixed thoroughly until all the powder form mixes with water to form solution of NaOH.

Preparation of Composite

The fabrication process for the composite material in this research study consists of four main steps: wetting, layup, consolidation, and solidification. Hand layup technique is used to fabricate the fibers, where the natural fiber is mixed with epoxy resin and a mild pressure is applied by a roller to remove any entrapped air. This technique is cost-effective, efficient, and durable. The resin and hardener ratio used in the process is maintained at 1:10. The schematic arrangement of the Sisal-Areca-Sisal fiber is illustrated in Figure 2 showing the layers of fibers in the composites during the hand layup fabrication process.

The fabrication process (shown in Figure 3) involves the preparation of a mold of a specific size (30mm x 30mm x 12mm Figure 3a), covering it with a transparent cover (Figure 3b), and applying wax (Figure 3c) on its surface to prevent the resin matrix fiber from sticking to the mold. Then, the hand layup stacking sequence is followed by adding the Epoxy resin Layer 1 (Figure 3d), Sisal Layer 1 (Figure 3e), Areca Layer (Figure 3f), and Sisal Layer 2 (Figure 3g) respectively. A roller is used to press the mixture so that the resin mixes with each corner and fiber placed in the mold, increasing the interfacial bonding between the base fiber and areca fiber. The mixture is then covered with a transparent plastic cover (Figure 3h), and a 10 kg load is kept on the mixture and left to cure for 6-8 hours. After curing, the material is marked with specimens for experimental testing. After curing process, the composite sample prepared looks alike as shown in the Figure 3i.

Sisal
Areca
Sisal

Figure 2. Stacking sequence sisal/areca/sisal composite material.

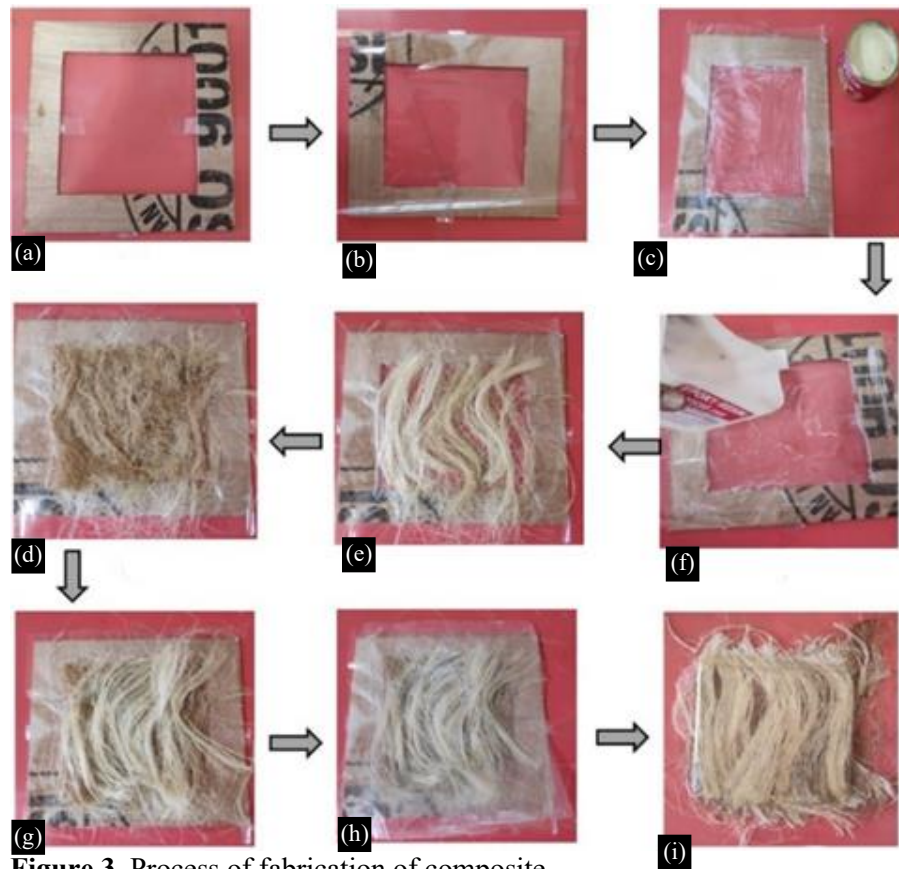


Figure 3. Process of fabrication of composite.

Table 2. Fiber Nomination and Orientation.

No.	Notation	Fiber	Description
1	USAS	Untreated Sisal-Areca- Sisal Based	Raw Sisal and Areca Fiber
2	SAS	NaOH treated Sisal-Areca-Sisal	Sisal and Areca fiber treated with NaOH treatment and then used for fabrication.
3	SASAL	Sisal-Areca-Sisal with fillers of Al ₂ O ₃	Raw Sisal and Areca fiber with Al ₂ O ₃ in powder form
4.	SASTI	Sisal-Areca-Sisal with fillers of TiO ₂	Raw Sisal and Areca fiber with TiO ₂ in powder form

Table 3. Fiber composition.

Fibers/materials	Material composition (%)			
	USAS	SAS	SASAL	SASTI
Sisal	15	15	15	15
Areca	12	12	12	8
Resin	73	73	65	70
Al ₂ O ₃	-	-	9	0
TiO ₂	-	-	0	7

EXPERIMENTAL TESTINGS

For conduction of experimental tests on the mechanical properties of the composite material, batches of three specimens were prepared for tensile, flexural, and impact strength measurements. The remaining cutout specimens were used for determining the density of the material. The description of the fibers used in the experiment are presented in Table 2 along with their respective notations and the composition of each material is illustrated in Table 3.

Tensile Test

Tensile test specimens (Figure 4a) are prepared using ASTM-638 [16] in which the specimen look alike dumbbell shape or dog bone shape as shown in Figure 4a. The tensile testing is performed on a universal testing apparatus set-TT-20 KN. In tensile testing, the specimen is grabbed in both the jaws and gradually the displacement in the jaws starts increasing thereby applying tensile force on the specimen and subjected to tension. The Elongation, ultimate tensile strength and yield strength of the materials are determined.

Flexural Test

The flexural test ASTM-790 standard is used to determine the flexural properties of reinforced and non-reinforced plastics including high modulus compounds and electrical insulating materials. The specimens are cut in rectangular (as shown in Figure 4b) following the ASTM-790. The flexural test is performed on an Ai-UTM 400 KN universal tester in which the specimen is kept on the 3 point at a distance of 50mm span length. The vertical tong is setup to bend exactly at the center of the specimen with creating the bending deflection and measuring the peak load when the specimen is broken. Hence forth the flexural strength was calculated by equation $[3PL/2bd^2]$ where p is the bending load, L=length of span, b is the width of specimen and d is the depth or thickness of specimen [16].

Impact Test

The impact test is performed according to ASTM D256 to enhance the impact resistance properties of materials viz. impact energy absorbed and thereby impact strength. Izod Impact test setup is been used to determine the same. The specimens are cut according to ASTM D256 standards (Figure 4c.) The specimen is kept vertical and the pendulum is released thereby impacting the specimen and breaking it. Henceforth the energy absorbed and impact strength is displayed digitally and been recorded.



Figure 4. a. Tensile specimen, b. flexural specimen, c. impact specimen. d. shore hardness specimen.

Density Test

The ASTM standard D792 is utilized as a general procedure for testing the density of plastics and polymers. To measure the weight of the specimen in different conditions, an analytical balance manufactured by Citizen (Make Model CG203) with a least count of 0.001 gram and a maximum weight measuring capacity of 200 grams has been employed. The primary aim of conducting this density test is to investigate the impact of alkalization, Al₂O₃, and TiO₂ on the weight of the specimen.

Shore Hardness Test

The test procedure conforms to ASTM 2240, which employs indentation of a loaded material. The test is performed using a durometer, which utilizes broken specimens (Fig. 4d) from flexural testing to create indentation marks, and the hardness is measured and recorded. The specimen's thickness falls within the range of 4mm to 7mm. The hardness tester comprises of an indenter, a pressure plunger, and a calibrated scale. The test is carried out by applying the pressure point on the specimen's surface, and the resulting hardness number is displayed on the scale.

RESULTS AND DISCUSSIONS

Figure 5a depicts the specimen's failure in the tensile test, while Figure 5b shows the failure in the flexural testing. The failure of the specimen in the impact test is illustrated in Figure 5c. Three composite samples were prepared for each test in batches, and the average value of each test is

presented in Table 4.

Table 4. Results of experimental testing.

No.	Properties of material	Composite material			
		<i>USAS</i>	<i>SAS</i>	<i>SASAL</i>	<i>SASTI</i>
1.	Tensile Strength (N/mm ²)	21.54	17.14	18.76	19.28
2.	Yield Strength (N/mm ²)	15.89	13.64	17.83	16.57
3.	Maximum Bending Load (KN)	0.35	0.18	0.86	0.61
4.	Flexural Strength (N/mm ²)	201.94	104.67	228.69	315.90
5.	Impact Energy Absorbed (J/cm)	73.83	125.37	161.67	317.57
6.	Impact Strength (J/cm ²)	1.74	1.93	2.50	5.90
7.	Density (g/cm ³)	1.07	1.35	1.86	1.60
8.	Shore D Hardness Value	84.67	87.33	83.33	89.00

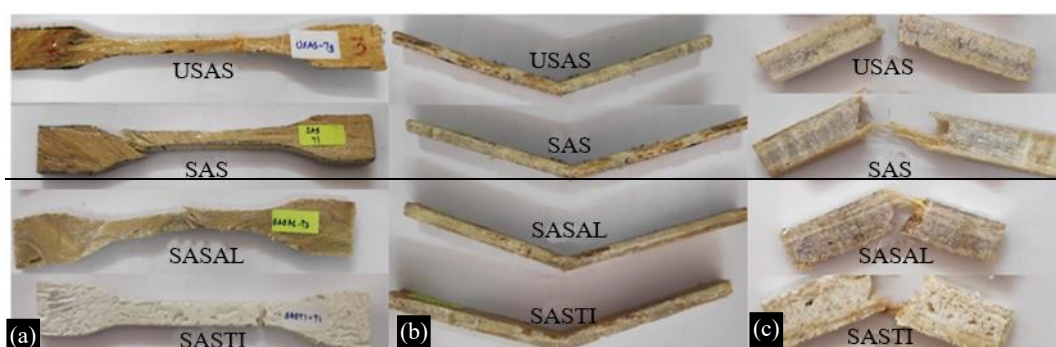


Figure 5. (a). Specimens of tensile Test (b). Specimens of flexural Test (c). Specimens of impact Test

Tensile Properties of Material

The tensile strength and yielding strength produced during the test are explained in the Figure 6 (a-b). The tensile strength produced in the Untreated Sisal-areca-Sisal composite produced maximum strength of 21.54 MPa and whereas the lowest is obtained for treated Sisal Fiber composite SAS with 17.14 MPa. The effect of adding filler of Al₂O₃ increases the tensile strength to 18.76 MPa and for TiO₂ it is obtained as 19.28 MPa. The results indicate that if the composite is added with fillers of Al₂O₃ and TiO₂ the strength can be increased by 8.6% for Al₂O₃ fillers and 11.09% for TiO₂ fillers. Figure 6b shows the typical comparison of difference in yield strength of sisal-areca composites treated with alkali or fillers. SASAL fiber with Al₂O₃ composite produced the highest yield strength with 17.83 MPa whereas Alkaline treated SAS composite produced lowest amount of strength with 13.64MPa. The SASTI fiber with TiO₂ filler showed intermittent yield strength with 16.57 MPa followed by untreated SAS with 15.89 MPa.

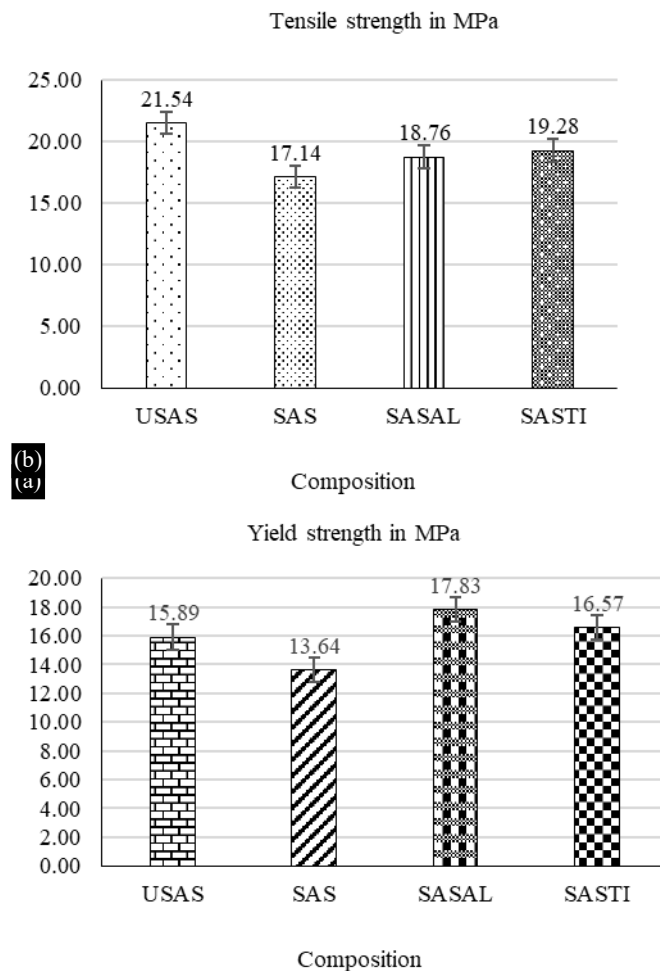


Figure 6. a. Ultimate tensile strength b yield strength.

Flexural Properties of Material

The SASAL fiber with Al₂O₃ absorbs maximum bending load of 0.86 KN (Figure 7a) whereas the alkaline treated SAS fiber fails at lowest bending load of 0.18 KN (Figure 7a). Figure 7b indicate the flexural strength produced in different combination of Sisal-Areca composites. The maximum flexural strength is obtained for SASTI fiber with 315 MPa (Figure 7b) and for alkaline treated sisal-areca SAS fiber it is obtained as lowest of 104.67 MPa (Figure 7b). The composite with Al₂O₃ filler, SASAL also show better result than USAS and SAS with 228.69 MPa for SASAL and for USAS it is obtained as 201.94 MPa. The outer fiber of the stacking sequence, fabrication bonding and thickness of the specimen plays vital role in flexural strength of the specimen.

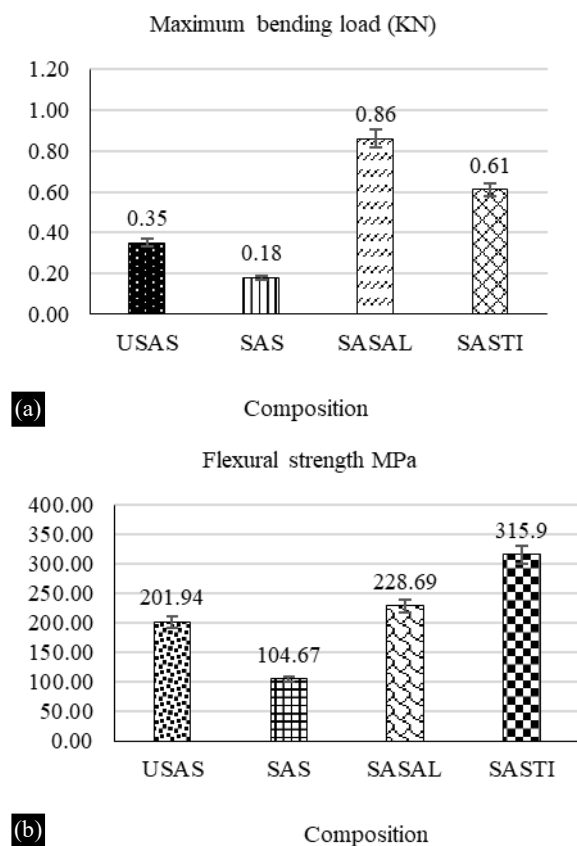
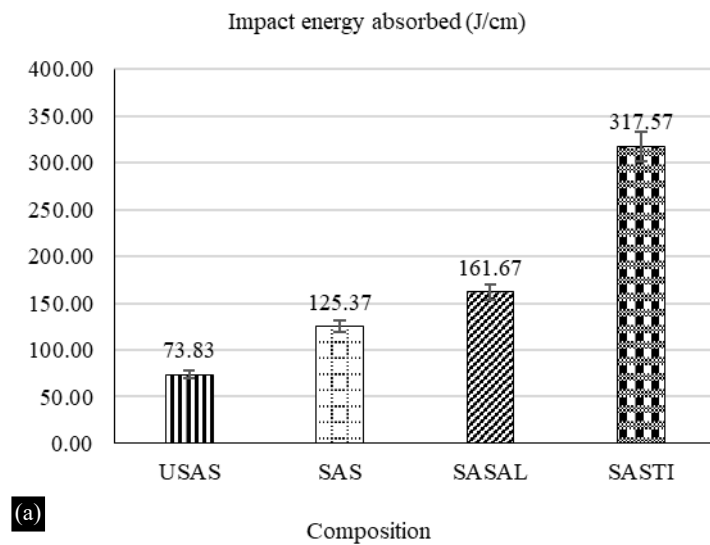


Figure 7. a) Maximum bending load b) maximum flexural strength
Impact Properties of Material

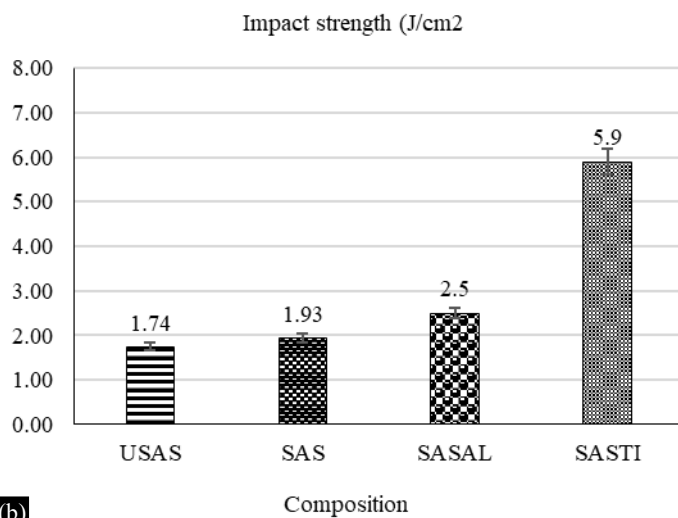
The absorption of energy increases as the fiber is treated with NaOH and obtained results as 125.37 J/cm for SAS. If the fillers of Al_2O_3 is added to composite, the energy absorbed for SASAL is obtained as 161.67 MPa and for fillers of titanium oxide, SASTI fiber shows the most energy absorbed amongst all with 317.57 MPa. The SASTI fiber displayed the best impact strength of 5.90 J/cm² whereas the untreated USAS fiber shows the lowest amount of impact strength of 1.74 J/cm². The fibers of SAS and SASAL showed the moderated impact strength of 1.93 J/cm² and 2.5 J/cm² respectively. The Figure 8a and Figure 8b, shows that effect of alkalization increases the impact strength of the material and whereas introducing fillers of Al_2O_3 and TiO_2 enhance more impact strength properties of the material.

Density of Material

The Maximum density (Figure 9a) was obtained for SASAL composite with 1.86 g/cm³ and the lowest was obtained for untreated USAS fiber with 1.07 g/cm³. The fibers of Alkaline treated SAS and Fiber with filler of TiO_2 , SASTI showed moderated density with 1.35 g/cm³ for SAS and 1.60 g/cm³ for SASTI. The comparison from the Figure 9a shows that the alkaline treatment increases the density of material as that of untreated composites, thereby the composite density increases more if they are added with fillers of TiO_2 and Al_2O_3 respectively.

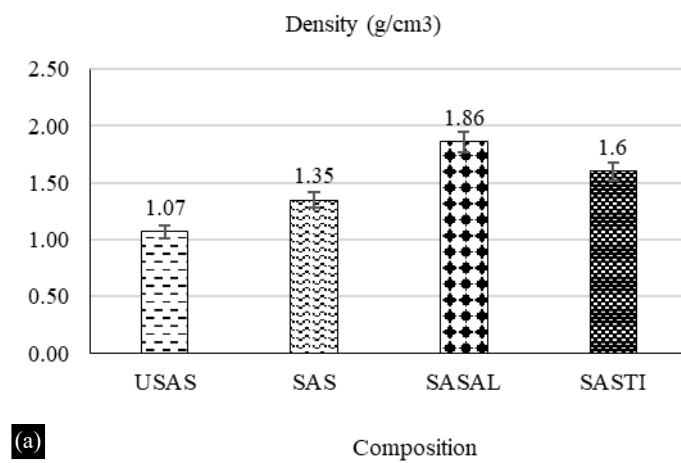


(a)

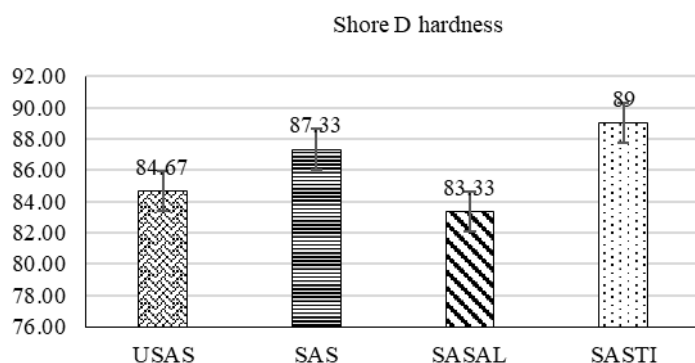


(b)

Figure 8. a. Impact energy b. impact strength.



(a)



(b)

Composition

Figure 9. a. Density of material b. shore D hardness of material.

Shore D Hardness of Material

The hardness value for each is measured and comparison of average value is explained in Figure 9b. The NaOH treatment resulted in an increase in material hardness by 3.04%. Specifically, the SAS composite achieved a hardness of 87.33 and the USAS composite achieved a hardness of 84.67. Upon adding TiO₂ filler to the Sisal-Areca Composite, the hardness increased by 1.89%, achieving a value of 89, which is higher than the SAS composite. However, when fillers of Al₂O₃ were added, the hardness value decreased by 4.8%, with the SASAL composite achieving a hardness of 83.33 compared to SAS.

CONCLUSIONS

- The composite that utilized raw sisal and areca fibers without any alkaline treatment exhibited the highest tensile strength, with the USAS composite reaching 21.54 MPa. In comparison, the treated SAS composite achieved a tensile strength of 17.14 MPa. The untreated USAS composite experienced a 20.42% increase in tensile strength.
- The addition of Al₂O₃ and TiO₂ fillers resulted in a gradual increase in tensile strength. When comparing the treated Sisal-areca fiber composite to SASAL and SASTI, the addition of Al₂O₃ increased tensile strength by 8.6%, and the addition of TiO₂ increased tensile strength by 11.09%.
- When using untreated raw material, the resulting fiber showed a 14.15% improvement over alkaline-treated SAS fiber. Additionally, adding Al₂O₃ fillers increased yielding strength by 22.93%, while adding TiO₂ fillers increased yielding strength by 17.68% compared to the treated SAS fiber composite.
- When adding Al₂O₃ fillers, the flexural strength of the composite increases by up to 54.23%, while the addition of TiO₂ fillers results in a 66.86% increase in flexural strength compared to alkaline-treated SAS. The SASTI fiber with TiO₂ filler exhibits 27.60% more flexural strength than that of Al₂O₃.
- The impact strength of the SAS fiber treated with NaOH alkaline solution is 9.88% higher than that of the untreated USAS composite. The addition of TiO₂ fillers increases impact strength by 67.28%, and the addition of Al₂O₃ fillers increases impact strength by 22.8% compared to alkaline-treated SAS fiber. Overall, it is observed that treating the fibers with alkaline NaOH produces a 9.8% improvement in impact strength.
- The addition of Al₂O₃ fillers increases density by 27.41%, while TiO₂ fillers in SASTI increase density by 15.62% compared to alkaline-treated SAS composite. Treating the fibers with NaOH increases material density by 20.07% compared to untreated USAS composite.
- NaOH treatment increases the material's hardness by 3.04%, and adding TiO₂ fillers to Sisal-Areca Composite increases hardness to 1.89%.
- In conclusion, the hybridization of composite material with Al₂O₃ and TiO₂ fillers improves the mechanical properties of the material but also increases the density of the composites.

- Sisal fiber can be used in various application including the structural and integral components of automobile applications where the required properties of materials are suitable.

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