



Examination of mechanical and morphological properties of jute/cotton natural fiber reinforced hybrid composites

Jüt/Pamuk doğal elyaf takviyeli hibrit kompozitlerin mekanik ve morfolojik özelliklerinin incelenmesi

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Abstract

Numerous studies are being conducted on composite products, the use of which is rapidly increasing due to their many superior properties. Manufacturers have tended to use natural fibers in composite making in order to prevent environmental pollution and increase the amount of raw materials. In this study, hybrid composites were produced by using vinyl ester resin as a matrix and natural fiber jute and cotton fabrics as reinforcement material. Manufacturing of composite samples was carried out by VARTM (Vacuum Assisted Resin Transfer Molding). The produced samples were examined mechanically by hardness, tensile, impact tests, and morphologically by scanning electron microscopy analysis. The lowest tensile strength value was 32.25 MPa in the reference cotton (RC) sample, while the highest value was 42.16 MPa in the reference jute (RJ) sample. In the impact test results applied to the samples, it was observed that the RJ sample had the highest impact energy with 51 J, while the lowest value was determined as 43 J in the sample RC. In addition, the water absorption properties of the samples were tested. While the highest water absorption value was found in the RC sample at 7.98%, the lowest water absorption value was observed in the RJ sample at 7.45%. On the other hand, it has been observed that jute and cotton fabrics interact well with vinyl ester resin. Finally, it has been the most important output of the research that the use of jute-cotton hybrid composites in many areas of industry, especially automotive, has high potential.

Keywords: Composite, Mechanical properties, Morphology, Resins, Biofibers.

Öz

Pek çok üstün özelliğinden dolayı kullanımı hızla artan kompozit ürünler üzerinde çok sayıda çalışma yapılmaktadır. Üreticiler, çevre kirliliğini önlemek ve hammadde miktarını artırmak amacıyla kompozit yapımında doğal elyaf kullanmaya yönelmişlerdir. Bu çalışmada, matris olarak vinil ester reçinesi ve takviye malzemesi olarak doğal elyaf jüt ve pamuklu kumaşlar kullanılarak hibrit kompozitler üretilmiştir. Kompozit numunelerin üretimi VARTM (Vakum Destekli Reçine Transfer Kalıplama) ile gerçekleştirilmiştir. Üretilen numuneler mekanik olarak sertlik, çekme, darbe testleri ile ve morfolojik olarak taramalı elektron mikroskobu analizi ile incelenmiştir. En düşük çekme mukavemeti değeri 32.25 MPa ile referans pamuk (RC) numunesinde, en yüksek değer ise 42.16 MPa ile referans jüt (RJ) numunesinde elde edilmiştir. Numunelere uygulanan darbe testi sonuçlarında RJ numunesinin en yüksek darbe enerjisine 51 J ile sahip olduğu, en düşük değer ise RC numunesinde 43 J olarak belirlendiği görülmüştür. Ayrıca numunelerin su emme özellikleri test edilmiştir. En yüksek su emme değeri %7.98 ile RC örneğinde bulunurken, en düşük su emme değeri %7.45 ile RJ örneğinde gözlemlenmiştir. Öte yandan, jüt ve pamuklu kumaşların vinil ester reçinesi ile iyi etkileşime girdiği gözlemlenmiştir. Son olarak jüt-pamuk hibrit kompozitlerin başta otomotiv olmak üzere sanayinin birçok alanında kullanım potansiyelinin yüksek olduğu araştırmanın en önemli çıktısı olmuştur.

Anahtar kelimeler: Kompozit, Mekanik özellikler, Morfoloji, Reçineler, Biyofiberler.

1 Introduction

Nowadays, material science has reached an important level with developing technology and intensive studies. Researchers working on material science basically examine solid materials in 3 classes metals and their alloys, ceramics, and polymers [1]-[3]. Composite materials, which are formed by the combination of more than one of these main materials, are considered the fourth main category [4]. Composite materials are materials that develop properties that are not present in each of the components by combining at least two different materials in such a way that they do not dissolve in each other [5]. Composites consist of a base material called a matrix and a durable material called a reinforcing element. In composite materials that are prevalently used in various engineering fields such as aviation, construction, automotive, and shipping, the most widely used matrix elements are polymers (polyester, epoxy, vinyl ester, polypropylene etc.). On the other hand, while

synthetic products (glass, carbon, aramid etc.) have stood out as reinforcement elements, researchers have focused on the use of natural fibers as reinforcement elements with the increase in sustainability sensitivity in the world in recent years [6]. In our world where technology is developing rapidly and industries are growing continuously, raw material shortage and environmental pollution are among the most critical problems. Under these conditions, it is important to use natural fibers in material production. Natural fibers are the preferred resource with their features such as being recyclable, degradable, easy to produce, and renewable, moreover, they are lower-cost raw materials [7]-[9]. Natural fibers are divided into three classes according to their origin plant, animal, and mineral-based fibers. Plant-based fibers are the most widely used among natural fibers and jute, cotton, flax, sisal, bamboo, and kenaf fibers can be given as examples of plant-based natural fibers [10]. Composite materials are highly preferred by researchers and manufacturers due to their lightness and easy design. The

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use of natural fibers instead of synthetic materials in composite materials gives these materials more environmentally friendly properties [11],[12]. In addition, natural fiber composites have less density, and biodegradability compared to synthetic fiber composites, which makes natural fiber composites stand out [13]-[16]. The ease of processability of natural fiber composites and their lesser effect on the health of the producer are among the other features that make these materials attractive. On the other hand, the disadvantages of natural fibers such as decomposition under room temperature conditions, sensitivity to water, and inconsistent fiber sizes should not be ignored. Therefore, it is more appropriate to use thermoset polymer matrices that can be processed at low temperatures and have a low melting point in natural fiber composites [7]. Since natural fiber fabrics have various positive and negative properties, manufacturers produce hybrid natural composites by combining different natural fibers, as they produce composite materials by combining different synthetic fibers [17]. In hybrid composites, various properties of the composite material are optimized according to the design requirements [18],[19]. To give an example, in designs where high strength is not required and the unit mass of the material needs to be reduced, low-density natural fiber fabrics can be used in the production of composite materials with different fabrics, or there are studies in which different fibers are used in the fabrication of various natural fiber hybrid composites to optimize the water absorption of the composite material [20]. In a study, Naveena et al. produced a natural fiber hybrid composite using polypropylene resin with natural fibers of flax, banana, and sisal. The researchers, who produced 4 different natural fiber composites in total as binary combinations and triple combinations of these natural fibers, examined the mechanical properties of the products. The researchers stated that the composite produced by using three different types of fibers together has the highest elastic modulus [21]. In another study, Cavalcanti et al. produced hybrid composites using fibers of jute, sisal, curaua and glass fiber. Researchers using epoxy resin, one of the thermoset resins, as matrix material in the production of composites, determined the fiber resin ratio as 30/70. Researchers, who included the alkalization process applied to the fibers in their research, applied tensile, impact, and flexural tests to the samples to examine the samples mechanically, and performed SEM (scanning electron microscopy) analysis to examine them morphologically. In the research, the results of the hybridization process were examined according to the pure jute composite. In the test results, it was observed that the composite strengths increased by 68% in the sisal and jute hybrid composite, 72% in the curaua and jute hybrid composite, and increased by 90% in the glass and jute hybrid composite compared to the jute composite. At last, the researchers stated that natural fibers are an important alternative to replace glass fibers [22]. Sathishkumar et al. produced a hybrid composite with a polyester matrix using sisal and cotton fibers. The researchers, who created a new design by changing the laying directions of the fabrics, examined the mechanical, morphological, and free vibration properties of the samples produced with different volumetric ratios. According to the results of the research, while the best mechanical properties were seen at 40% by volume, it was observed that the differentiation of the fabric laying directions had a positive effect on the mechanical properties of the material [23]. Giridharan et al. produced a new hybrid composite with epoxy resin by combining cotton and glass fibers. The samples were produced with 20% and

30% fiber ratios and were subjected to impact tests, flexural tests, tensile tests, and SEM analyses. The hybrid product was compared with composite samples made of cotton only and glass fibers only. As a result, the researchers stated that the product with a fiber content of 30% by weight had higher strength, while the hybrid products were also superior to the others [17]. Masood et al. produced a hybrid composite with polyester matrix using cotton, jute, and glass fibers. The researchers, who produced three reference samples (100% cotton, 100% jute, 100% glass) and four different hybrid composites in which different proportions of fibers were combined, applied mechanical and morphological analysis to the samples. In the results of the research, it was explained that the strengths of cotton and jute composites were lower than glass fiber, but hybrid composites had close strength to glass fiber composites [24]. de Carvalho et al. produced a polyester matrix composite using cotton-jute and cotton-sisal hybrid structures. The fabrics were first immersed in water, then dried before production, and investigated how these processes affect the tensile strength of composite products. While the researchers explained that sisal fiber retains more water than jute fiber, they expressed that pre-drying improves the mechanical properties of the material. Besides, it has been shown in the research results that the pre-production water immersion process does not affect the jute fabric, but negatively affects albeit to a small extent the mechanical properties of the composite produced with sisal fabric [25]. Ramprasad et al. produced composites with cotton waste-jute and cotton waste-glass fiber hybrid combinations using epoxy resin (65% by weight). Researchers, who added 10, 15, 20, and 25 percent glass and the same proportions of jute fiber to cotton dust waste, also included the alkalization process and produced 10 different samples in total. Researchers performed mechanical tests (tensile, impact and flexural test) on the samples. In the research results, it is expressed that alkali treatment improves the mechanical properties of the material; it has been explained that cotton dust waste/jute hybrid composite has higher tensile strength, flexural strength, tensile modulus, and cotton dust waste/glass hybrid composite has higher impact strength and flexural modulus. Eventually, the researchers added to their explanation that cotton and jute natural fibers are an important alternative to glass fibers [26]. Alsina et al., in order to investigate the water absorption behavior of natural fibers, produced polyester-based composites by hybridizing ramie, jute and sisal fibers with cotton. The researchers, who produced the composite by immersing the fibers in water and drying them, also investigated the effect of immersion temperature on the water absorption properties of the samples. In the results of the research, it was stated that immersion temperature is not an important parameter in water absorption, and the main parameter affecting this feature is the volumetric ratio of the fibers in the product. Lastly, they explained that jute/cotton and ramie/cotton hybrid composites are more hydrophobic than sisal/cotton hybrid composites [27]. In this study, a hybrid polymer matrix composite was produced with different combinations of two different natural fabrics as reinforcement agents and by using vinyl ester as a matrix element, which is a thermosetting resin. The specimens were mechanically examined by being subjected to tensile, impact and hardness tests. Besides, the investigation of the fracture structure of the composite material by SEM analysis was included in the research. Finally, the moisture retention of the material was examined by applying the water absorption test to the produced samples.

2 Experimental

2.1 Materials

The natural fiber fabrics used in this research were jute and cotton. The reasons for choosing these fabrics are that they are inexpensive, suitable for hybridization, less hydrophilic than other natural fibers, and easy to find. The purpose of hybridizing these two natural fibers is to optimize material strength and hydrophilicity. Fabrics purchased from a local firm in Adana, Turkey is shown in Figure 1 and fabrics properties are given in Table 1.



Figure 1. Jute and cotton fabrics.

Table 1. Fabrics properties [10].

Fabric	Weight (gr/m ²)	Thickness of Fabric (mm)	Warp (tex)	Weft (tex)
Cotton	150	0.2	–	–
Jute	265	0.7	312.5	312.5

The vinyl ester resins chosen as the matrix material in this study are a class of resins that combine the best properties of both polyester resins and epoxy resins in their structure. While they have high mechanical strength values like epoxy resins, they offer easy processing like polyester resins. The most important features are mechanical and chemical resistance and heat resistance [28],[29]. The vinyl ester enters into a chemical reaction by mixing with methyl ethyl ketone peroxide (hardener) 2% of resin (by weight) and cobalt (II) 2-ethyl hexanoate (accelerator) products 0.2% of resin (by weight), it cures and takes the shape of the mold. Table 2 indicates the technical properties of the vinyl ester resin.

Table 2. Vinyl ester properties [10].

	Molding	Laminate
Heat distortion temperature (°C)	137	-
Flexural strength (MPa)	130-140	132
Bending coefficient (MPa)	3400-4000	7400
Viscosity (mPas)		180-220
Tensile strength (MPa)	69-79	93
Barcol hardness	42	50

2.2 Fabrication of samples

The becoming of air gaps in the fabrication of composite materials is one of the most important parameters that reduce the quality of fabrication. The method that minimizes this problem is VARTM. VARTM is also very suitable for serial fabrication. This process is aimed to feed the resin to the fiber fabrics stacked in the vacuum package with the absolute pressure force, and after the process, the product is cured [30]. Therefore, this composite fabrication method was preferred in this investigation. The model of the VARTM system and prepared samples are shown in Figure 2.

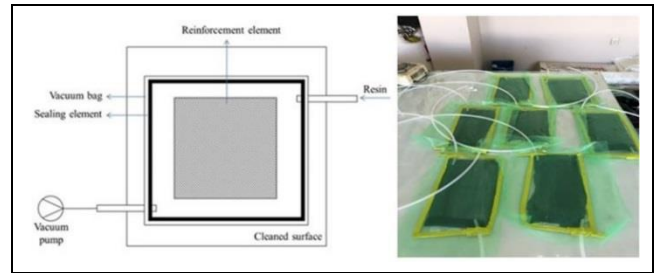


Figure 2. VARTM method schematic view and prepared samples.

In order to carry out fabrication, cotton and jute fabrics were placed in different sequences and various combinations were created. The fabrics were used without any chemical treatment and as received from the supplier. Then the system and vacuum package were prepared. To obtain a homogeneous mixture, the vinyl ester and accelerator were combined and mixed and the resin was obtained. Thereafter, the hardener chemical was incorporated and ten more minutes were mixed. After acquiring the homogeneous mixture, the resin was transferred into the vacuum package by running the system. Samples were cured at room temperature for twenty-four hours and whereupon extracted from the molds. After curing the samples for 1 hour in the oven at 60 °C, they were cut off in the test sizes designated in the norms. For the comparisons to be made to give more accurate results, reference samples were produced and these samples are 2 pieces, consisting of only cotton and only jute fabrics. Hybrid composites were produced in 5 different combinations and the superiority of the products to each other was compared. The samples consist of 6 layers and 12 layers of fabric, and the samples with 12 layers of fabric are the double versions of 6 layers of fabric. The samples were produced by the test conditions made within the scope of the research. The configurations of the produced samples are seen in Figure 3. The Fiber/matrix ratio of the RC sample is 0.66 by weight, while the Fiber/matrix ratio of the RJ sample is 0.24. While this ratio was 0.345 in samples C2 and C4, where jute fabric was dominant, it was 0.555 in sample C3, where cotton fabric was dominant. In samples C1 and C5, which are combinations with equal numbers of jute and cotton fabrics, this ratio was 0.45.

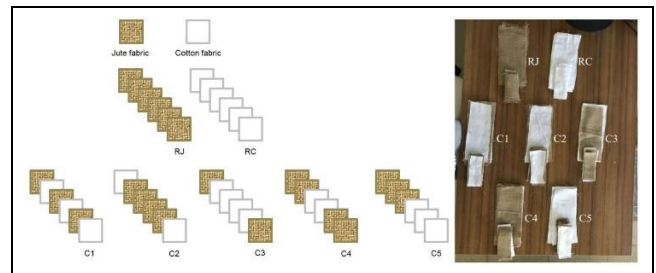


Figure 3. Sample configurations.

2.3 Tests of samples

After fabrication, specimens were mechanically analyzed by being subjected to tensile, impact, and hardness tests. The dimensions of tensile and impact test samples are 250 mm x 25 mm x 2.5 mm and 125 mm x 12.5 mm x 10 mm, respectively, according to related standards of tests [31],[32]. Tensile tests were performed with an ALSA hydraulic (60 tons) mechanical test device in the MTSO Accredited Test Center, and impact tests were performed with a charpy tester. The hardness data

of the samples were acquired with the Vickers micro-hardness test in Çukurova University Automotive Engineering Laboratory. 10 seconds dwell time and 0.3 kgf of the load were applied for these tests. The fracture structures of the specimens were examined at the micro-level by SEM (FEI Quante 650 FEG scanning electron microscope) in Çukurova University Central Research Laboratory. Finally, in order to calculate the water absorption capacity of the samples, the water absorption test was performed based on the ASTM D5229 standard. Samples smaller than the dimensions specified in the standard were used (20mm x 20mm). The samples produced, 5 of each configuration, were sealed with resin so that they did not absorb water from their sides. The samples were dried in an oven at 60 °C for 10 hours and then brought to room temperature. After this pre-treatment, the samples, which were immersed in water and dried, were weighed once an hour for the first 6 hours, after which they were kept at intervals of 18, 24, and 72 hours, respectively. The weights of the samples, which were kept in water for a total of 120 hours, were measured with an accuracy of 0.1 mg [27]. Using these

measurement results, the water absorption percentage (WAP) of the samples was calculated with the Equation (1):

$$WAP = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

Where W_f is the weight subjected to immersion and W_i is the weight before immersion. The average of 5 samples was taken and written in the equation. Then, the results were reported.

3 Results and discussion

3.1 Tensile test results

To determine the mechanical properties of the experimental samples, tensile tests were conducted to tests were performed three times, and the results were averaged. Figure 4, Figure 5, and Figure 6 denoted tensile strength, elongation, and modulus of elasticity values of materials with change percentages in tensile test results of hybrid arrangements (C1, C2, C3, C4, and C5) with respect to reference samples (R) and RC).

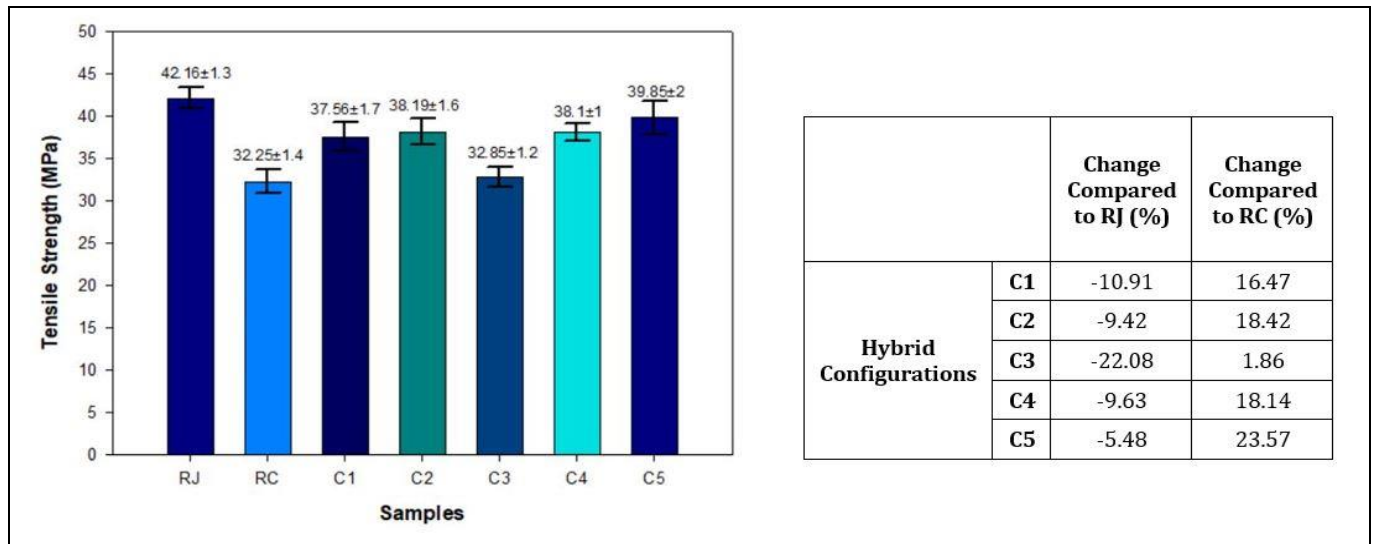


Figure 4. Tensile strength values of samples and change percentages of tensile strength with respect to reference samples.

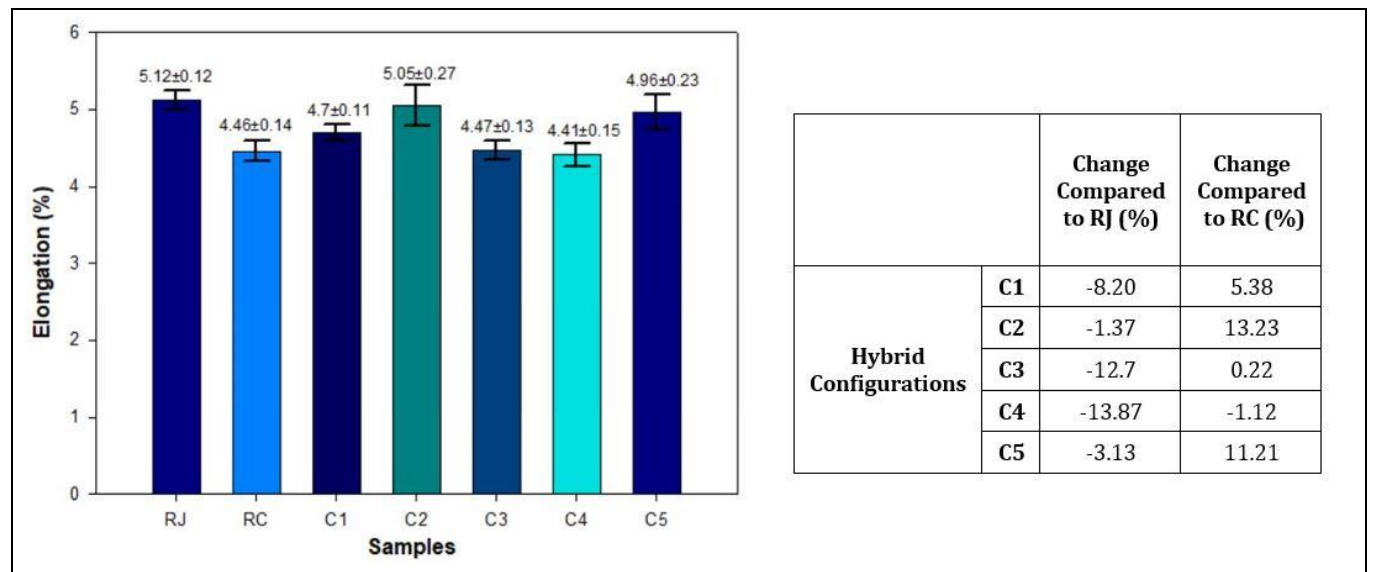


Figure 5. Elongation values of samples and change percentages of elongation with respect to reference samples.

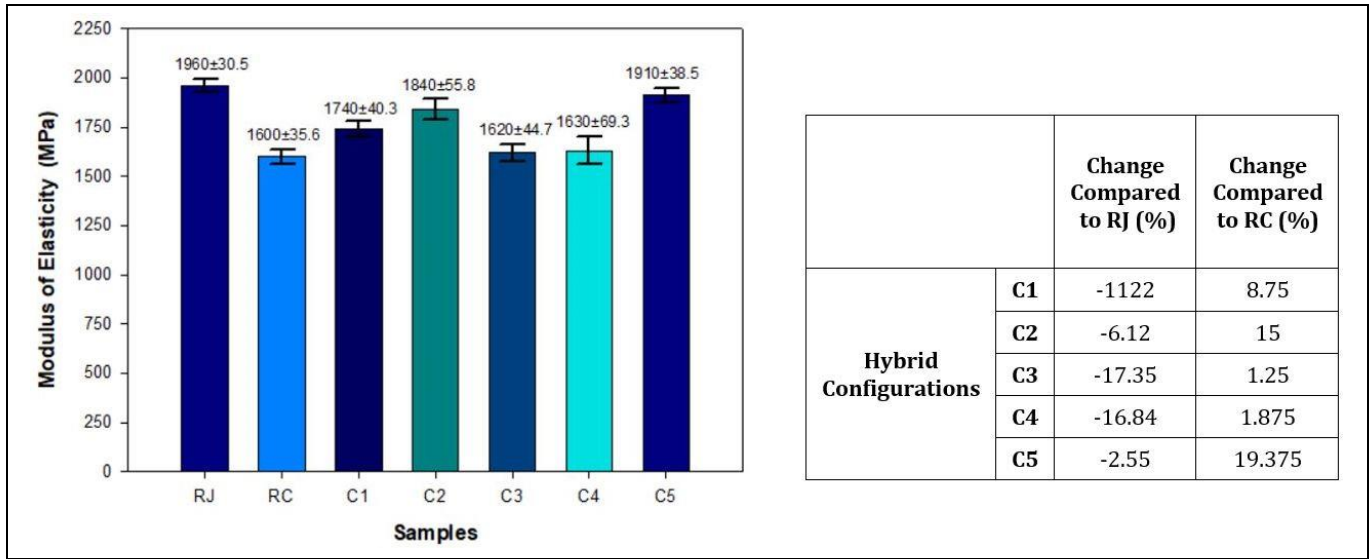


Figure 6. Modulus of elasticity values of samples and change percentages of modulus of elasticity with respect to reference samples.

The minimum tensile strength value was obtained for RC as 32.25 ± 1.4 MPa while the maximum was 42.16 ± 1.3 MPa for RJ. Tensile strength values of all hybrid arrangements lay between RC and RJ. The tensile strength of all hybrid configurations was scaled up compared to RC and deteriorated compared to RJ. Among the hybrid configurations, the highest tensile strength was observed in the C5 sample with 39.85 ± 2 MPa. The C5 sample has 5.48% lower tensile strength than the RJ sample and 23.57% higher tensile strength than the RC sample.

Elongation value is an important parameter that shows the ductility of the material. According to the test results, the highest elongation at break was observed in the RJ sample at $5.12 \pm 0.12\%$, while the C4 sample had the lowest value at $4.41 \pm 0.15\%$. The percent elongation at break in the C4 sample was observed as 13.87% compared to RJ. A much higher elongation at break was found in the C4 sample, which has the same number of jute and cotton fabrics as the C2 sample.

The minimum modulus of elasticity (MOE) value was observed for RC as 1600 ± 35.6 MPa while the maximum was 1960 ± 30.5 MPa for RJ. The modulus of elasticity values of all hybrid configurations lay between RC and RJ. The highest MOE among the hybrid samples was observed in the C5 sample. The C5 sample has only 2.55% lower value than RJ; it had 19.375% higher MOE than RC.

3.2 Impact test results

The impact test findings of the composite samples are shown in Figure 7. The results show that the cotton fiber-reinforced reference sample had lower impact energy than jute reinforced reference sample. With the hybridization, the impact energies were within the range of reference samples of RJ and RC. According to impact test results, the hybrid samples show higher impact energy when the jute reinforcement is dominant. The highest impact energy was found as 51 ± 2.7 J when the reference sample of jute (RJ) was tested. The impact energy of the cotton sample (RC) with the lowest value was determined as 43 ± 3.5 J.

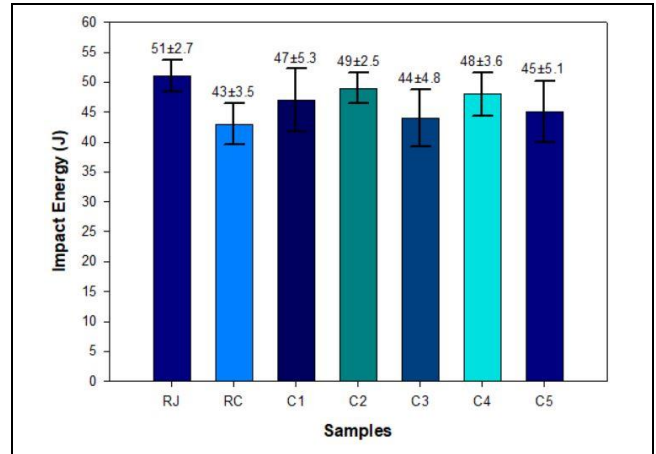


Figure 7. Impact test results of the composite samples.

Among the hybrid samples, the highest impact energy was observed in the C2 sample. The effect of laying jute fabrics, which are more durable than cotton fabric, in the middle part, attracted attention as a result of the impact test. While it was concluded that the C2 sample had 12.2% higher impact energy than RC, it was observed that it had only 4.08% lower impact energy than RJ.

3.3 Hardness test results

The Vickers hardness test results of the samples are shown in Figure 8. The results revealed that the combination of the fibers laid up has no significant effect on the samples' surface hardness. It was seen that the hardness values are dominated by the matrix material and there was a slight matrix layer on all composites that covers the fiber materials. Therefore, the hardness values of all composites were similar to each other.

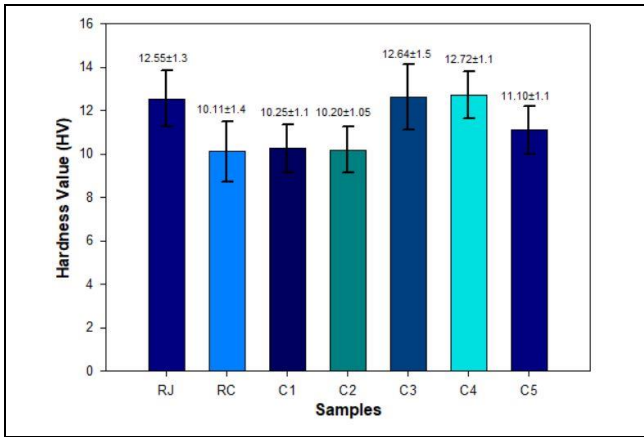


Figure 8. Hardness test results of the composite samples.

3.4 Water absorption results

The water absorption test explains the hydrophilic character of the structure. The water retention of composite products produced with natural fibers affects the deterioration time of the material. The water absorption test was applied to the samples as stated in the previous sections, and the test results are shown in Table 3 and Figure 9.

Table 3. Water absorptions of samples.

Time (h)	Water Absorption (%)*						
	RJ	RC	C1	C2	C3	C4	C5
1	2.36	2.95	2.88	1.93	2.08	1.79	1.74
2	4.17	3.81	4.01	3.67	3.63	3.21	3.22
3	4.67	4.75	5.11	4.94	5.34	4.79	4.31
4	5.01	5.75	6.19	5.76	5.80	5.44	5.59
5	5.75	6.33	6.94	6.41	6.58	6.37	6.10
6	6.23	7.16	7.25	6.74	6.97	6.75	6.81
24	6.48	7.35	7.50	7.34	7.44	7.24	7.45
48	7.07	7.78	7.69	7.45	7.67	7.47	7.64
120	7.45	7.98	7.82	7.62	7.90	7.65	7.84

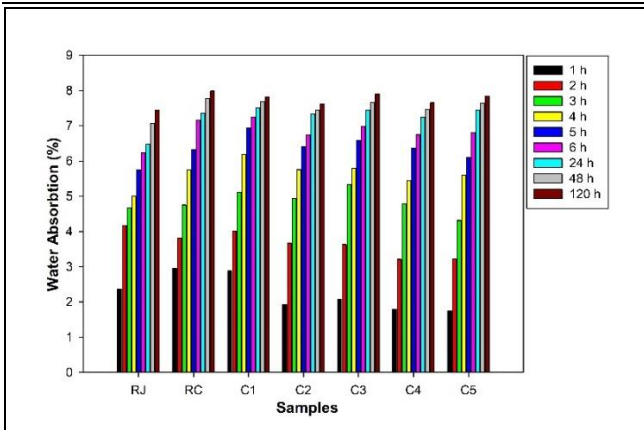


Figure 9. Graphic of water absorptions of samples.

When the results were examined, the highest water absorption value was found in the RC sample at 7.98%, while the lowest water absorption value was observed in the RJ sample at 7.45%. Among the hybrid combinations, the highest value was observed in the C3 sample with 7.90%, while the lowest value was observed in the C2 sample with 7.62%. Considering these values, it is understood that the water absorption capacity is higher in the samples with cotton fabrics in the majority, while it is observed that the values decrease in samples with dense jute fabric. There was no obvious difference between the

samples with the same number of fabrics and different sequences.

3.5 Scanning electron microscopy analyses

SEM analyses enable the microstructure and fracture mechanisms of the samples to be examined and contribute to their evaluation. Within the scope of this study, after the tensile test was applied to the samples, SEM images were obtained from the broken surfaces of the test samples. SEM images taken from the samples are seen in Figures 10 and 11.

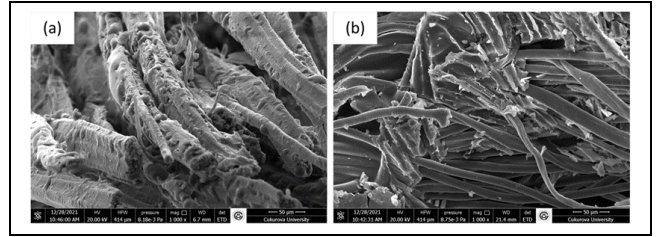


Figure 10. After the tensile test 1000x SEM image of (a): RJ. (b): RC.

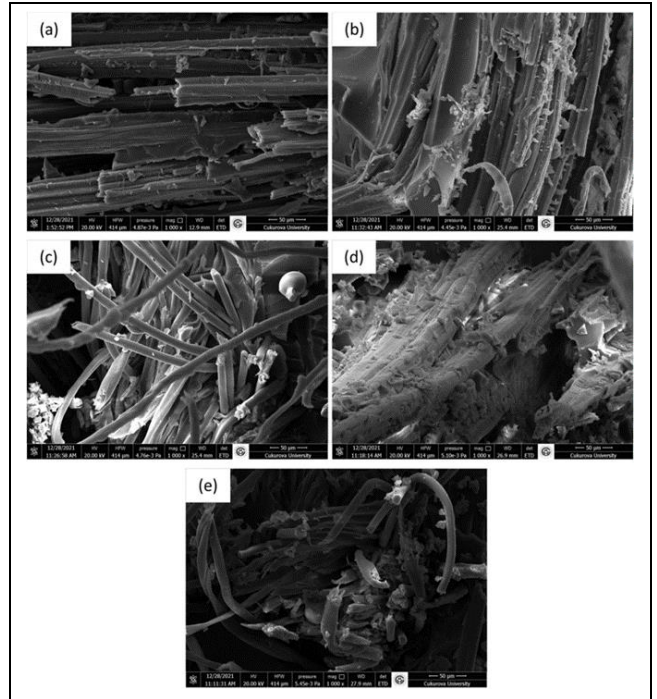


Figure 11. After tensile test 1000x SEM images of (a): C1. (b): C2. (c): C3. (d): C4. (e): C5.

It is figured out from Figure 10 that jute fibers form a more compact structure with the solidified resin. The higher tensile strength and impact energies of reference jute samples and jute dominant arrangements can be explained by better compatibility. In addition, the higher strength observed in the samples in which the jute fabrics are arranged one after the other can be attributed to this good interaction. SEM images show that cotton fibers fracture more elastically than jute fibers. While fibril elongations were seen when breaking in cotton fibers, sharp structures appear after breaking in jute fiber. The lower interphase strength of cotton and matrix material resulted in lower tensile and impact strengths.

Fiber fractures, strains, and bucklings can be understood from SEM images. Most of the failures occurred due to fiber breaks. When fiber breaks are examined, it is seen that interphase

adhesion is good, especially in jute fibers; However, it was understood from the SEM images that the interphase adhesion strength of cotton fibers was lower compared to jute fibers. According to the tensile test results, it was determined that jute fiber-based samples showed higher tensile strength, and their better mechanical performance thanks to their better interaction with the matrix material was also supported by SEM images.

4 Conclusion

In this research, a new type of hybrid composite was produced by combining jute and cotton natural fiber fabrics with vinyl ester, which is a thermosetting resin. While the number of fabric layers remained constant in the hybrid composite samples, which were handled mechanically and morphologically, the fabric sequences and jute-cotton content were changed and the effects of these changes on the product properties were observed. The results showed that hybrid composites with high jute fabric content have superior properties in terms of mechanical strength. In addition, due to the lighter weight of the cotton fabrics, it has reduced the weight of the samples and it has been observed that the strength of the material is only negligible. It has been observed that jute fabric stands out in terms of absorbed impact energy. In vehicle security elements, the use of hybrid combinations with high jute fabric content should be considered. Since the outer surface of the produced composites is mostly vinyl ester resin, stable results could not be obtained in the hardness tests. When the fracture structure of the materials is examined in SEM analysis; it has been observed that jute fibers form a more compact structure with resin. This explains that the samples with higher jute fiber content have higher mechanical strength. Cotton fabrics are more hydrophilic than jute fabrics. In line with this, another output of the results was that the hybrid combinations with higher cotton fabric content absorb a higher amount of water. Finally, the effects of fabric sequences on the material are also explained in the results of the study. Based on the results, hybrid composites designed with jute and cotton fabrics has a high potential to be used in interior structures in the aviation, automotive and construction sectors. Especially, in automotive sector, this new designed hybrid composites would be used at buffer sections. It is predicted that hybrid designs produced with these natural fiber fabrics will become more popular thanks to the flexibility they provide to designers and manufacturers.

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6 Contribution statements by authors

In this study, Şafak YILDIZHAN contributed to the creation of the idea, concept design, and data collection, Ayperi Tuğçe TOSUN contributed to the literature review, data collection, and production of samples; Mustafa Atakan AKAR contributed to the literature review, data analysis, and critical review, Umut KUMLU contributed to data analysis, writing and manuscript control sections.

7 Approval by the ethics committee and a conflict of interest statement

“There is no need to obtain permission from the ethics committee for the article prepared”.

“There is no conflict of interest with any person/institution in the article prepared”.

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