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The investigation of stearic acid treatments on the water absorption, cataplasm and pull off test of flax fiber/polyester composites

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Abstract: *Flax fiber is a reinforcement material which is weak against water because it is one of the natural fibers. To improve this weakness, it was treated with stearic acid (SA) at different ratios (1, 2, 3 and 4 %). In addition, the effect of gelcoat and paint application on the performance of the sample was also examined. Flax fiber takes on a hydrophobic structure when treated with different ratios of stearic acid. When the test results were analyzed, the lowest water absorption results were obtained with 4 % SA treatment. It was observed that the water absorbing properties of the composite improved with the application of gelcoat and paint. In the cross-cut test performed after the cataplasm test (aging test), the adhesion performance of none of the specimens was adversely affected. There was no blister observed after aging test. According to the pull off test results, stearic acid application did not have a negative effect on the adhesion of the primer and paint applied to the surface. After the cataplasm test, the specimens were aged and curing was thus complete. For this reason, pull off test results were higher after cataplasm.*

Keywords: *Flax fiber; Stearic acid treatment; Water absorption; Cataplasm; Pull off.*

1. Introduction

Most of the time, when composite materials are considered, the first thing that comes to mind is synthetic fiber reinforced composite materials. Due to the deterioration of the life conditions in our world day by day, the production towards natural and sustainable products has increased in many applications. The composites industry is one of the sectors that have started to use natural fibers. The use of natural fibers for composite materials is increasing day by day because they are renewable, cheap and recyclable. Natural fibers are classified as leaf fibers, seed fibers, bast fibers and grass fibers. Jute, flax, hemp, sisal, cotton are examples of natural fibers. There are some advantages of using natural fiber. The main advantages are environment-friendly, sustainable, cheap and easy to find. Beside these advantages, there are also some disadvantages. Some of its disadvantages are its poor interface and high tendency to absorb water. Low thermal stability and quality differences are also effective features on the final product. Natural fiber reinforced application areas can be transportation, military applications, building and construction applications, etc. [1-3].

Gelcoat application is one of the applications that is generally used in composite materials. Gelcoats can be epoxy or polyester based and they are referred to as modified resins. They can be applied by brush or spray. Gelcoat application is mostly realized to make the surfaces of the produced parts smooth. It is also used to protect the fibrous structure against external impacts. Except this, another reason is that it acts as a barrier and reduces take humid ability feature of the material [4,5].

Paint application is one of the largest processes for automotive applications. Paint is an organic coating with pigment (coloring agent) in its composition. The composition of the paint includes pigments, binders, additives and solvents. Raw materials and their ratios may vary depending on the place and purpose of use. The type of pigmented coating applied first to the surface on which the paint will be applied is called primer. The reasons for using a primer are: good adhesion, improvement of paint performance, ability to protect metal surfaces from rust, wood surfaces from mold and rot, having a covering to hide the appearance of the application surface, having the feature of good adhesion of the layers to be applied, well-spreading and be easy to sand [6]. The purpose of using paints and coatings is to protect the final product from environmental factors and adding aesthetic appearance [7].

Being hydrophilic structure is one of the biggest disadvantages of natural fiber. Due to its hydrophilicity, the fiber swells and decays. Some processes are realized to improve the fiber-matrix interface and add properties to natural fibers [3]. These processes can be classified as chemical or physical. For example, corona, plasma, and mercerization can be given. However, these processes can be a bit expensive in cost. Chemical treatments are generally preferred for surface improvement. The treatment of natural fiber with stearic acid is also one of the chemical surface improvement processes. The reason for using stearic acid is to replace the surface of

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the natural fiber with fatty acids. The working principle of the mechanism is as follows: The carboxyl group found in stearic acid reacts with the hydroxyl group found in natural fiber. Thus, a decrease in the number of hydroxyl groups occurs. It also removes impurities such as lignin and pectin on the fiber surface and creates a good fiber-matrix interface [8-10]. Chemical reaction of stearic acid treatment effect on natural fiber is shown in Fig.1.

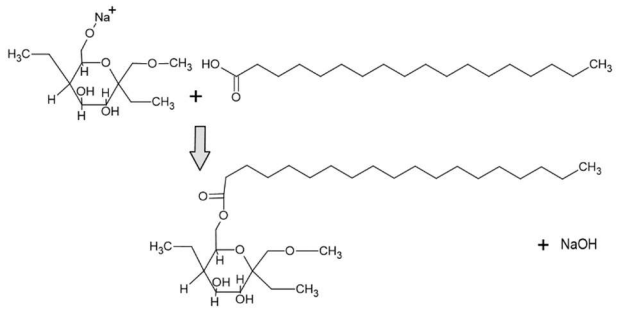

***Fig.1**. Chemical reaction of stearic acid with a natural fiber [11]*

Salem et al. [8] studied stearic acid treated kenaf fiber at 0, 0.4, and 0.8% ratios. They found that the water absorption rate decreased as the stearic acid treatment rate increased. Sreenivasan et al. [11] treated the fibers with alkali, benzoyl peroxide, potassium permanganate and stearic acid to improve the interface of cylindrical fibers with polyester. As a result of the study, it was concluded that chemically treated cylindrical fibers reached higher strength. An improvement in the water absorption properties of natural fibers was observed with chemical treatments. Jain et al. [12] evaluates the effects of mercerization and stearic acid treatment on the fibers. They observed a decrease in water absorption values with 4% stearic acid treatment applied to palmyra fiber. Dolez et al. [13], studied hydrophobic treatments of natural fibers. They treated the natural fiber with titanium dioxide, zinc oxide and stearic acid. After the stearic acid treatment, the contact angle of the samples was measured. While the contact angle in untreated jute fiber was 77°, after the treatment, it becomes 120°. This indicates that jute fiber has gained hydrophobic character.

Kiattipanich et al. [14] studied stearic acid treatment of sugarcane fiber reinforced polypropylene composites. The moisture content was investigated in this research. They dissolved different proportions (3, 5, 7 and 9%) of stearic acid in ethanol. Then the fibers were immersed in this solution and dried. When the composite produced, it was observed that sugarcane fiber treated with stearic acid retained less moisture compared to the untreated fiber. The aim of this study is to improve the surface of flax fiber, which has high moisture absorption ability, by treating it with stearic acid. The objective of this study is to observe the effect of gelcoat, primer and paint on moisture absorption behavior of flax fiber/polyester composite materials.

2. Material and methods

2.1. Materials

300 g/m^2 plain woven flax fabric was purchased from B-Preg (Turkey). Unsaturated polyester resin used for a matrix. 2 % Methyl ethyl ketone peroxide (MekP) used for hardener of polyester resin according to technical data sheet. The epoxy-based primer and acrylic based basecoat are also used. Primer, paint and isopropyl alcohol were bought from DYO. For surface treatment processes, stearic acid (SA) was purchased from Bursa Teknik Kimya. Whole material and equipment required for the production and process were provided by SAZCILAR A.S.. All productions were realized at room conditions (23±2°C and Relative Humidity 50 ±10%).

2.2. Surface treatment of flax fabrics

In this study, flax fibers were soaked in ethanol at different weight ratios (1, 2, 3 and 4%) of stearic acid solution. To compare the effect of stearic acid treatment, one group of flax fibers was untreated. As Jain et al. [12] did in their study, the stearic acid was mixed in a mechanical mixer at 30 °C from the moment it was added to the ethanol until it was mixed. The aim of the applied temperature is to make the mixture easily. The fibers were soaked in the solution for approximately 30 min. Then, the fibers were removed from the solution and the water was allowed to drain. The filtered fibers were left to dry in a 100°C oven for approximately 1 h. At this step, white spots were observed on the fibers as the concentration of stearic acid in the solution increased. Chemically treated flax fibers were stored in a dark place.

2.3. Production of treated laminated composites

In order to see the effect of the materials used in production, the experiment was divided into 3 groups:

2.3.1. Production of non-gelcoated flax fiber/polyester composite treated with stearic acid

At this stage, the flax fibers were dried in an oven at 60 °C for 1 h before proceeding to the production step after stearic acid treatment in order to remove moisture. After this step, a 40x50 cm mold was used for production. Flax fibers were cut in 25x40 cm dimensions. In order to remove the produced test plate from the mold easily and to avoid to stick the surface, mold release chemicals were applied. Mold release agent was applied to the mold surface 3 or 4 times and then wiped with a clean cloth. After the mold preparation phase was completed, the mold surface was wetted with polyester by using a brush. Flax fiber was laid on the mold surface and passed over it with a roller. The laid fiber was wetted with polyester again and this process continued until the production of 3 layers of flax fiber was completed. After the flax fiber untreated with stearic acid was produced by hand lay-up method, the simultaneously 1, 2, 3, and 4% stearic acid-treated composite plates were produced by the same production method. After the production step was

completed, curing was carried out at room conditions (23±2°C and Relative Humidity 50 ±10%) for 24 h and at 50°C for 1.5 h. Finaly, the samples were cut in the standard test sample sizes.

2.3.2. Production of gelcoated flax fiber/polyester composite treated with stearic acid

At this stage, the flax fibers were dried in an oven at 60°C for 1 h before proceeding to the production step after stearic acid treatment in order to remove moisture. After drying, a 40x50 cm mold was used. In this step, gelcoat was used to get smooth part surface appearance. It is also used to protect the fibrous structure against external impacts. Gelcoat was applied according to technical data sheet and it was expected to reach dryness to the touch. The spray gun has a 1.3 mm nozzle and the application was performed with a pressure of 3.5-4 bar. When the gelcoat was achieved the tack-free time for production, composite panel was produced by hand lay-up method again. The production stage and curing process were carried out in the same way as the untreated flax fiber composites and the samples were cut in the standard test sample sizes.

2.3.3. Production of gelcoated and painted flax fiber/polyester composite treated with stearic acid

At this stage, as in the other stages, the flax fibers were dried in an oven at 60°C for 1 h after the stearic acid treatment, before proceeding to the production step, in order to remove moisture. After drying, a 40x50 cm mold was used. In this step, gelcoat was used to get smooth part surface appearance. It is also used to protect the fibrous structure against external impacts. Gelcoat was applied according to technical data sheet and it was expected to reach dryness to the touch. The spray gun has a 1.3 mm nozzle and the application was performed with a pressure of 3.5-4 bar. When the gelcoat was achieved the tack-free time for production, composite panel was produced by hand lay-up method. The production stage and curing process were carried out in the same way as the untreated flax fiber composites and the samples were cut in the standard test sample sizes.

In addition to the other steps, paint was applied to the gelcoat surfaces of the samples prepared in the size of the test sample. Surface preparation is important for primer and paint application because it increases the roughness on the surface and facilitates adhesion [7]. Firstly, the gelcoated surface was sanded to get good adhesion between paint and gelcoated surface. Then, compressed air was used to remove the dust on the part surface. Next, the part was cleaned with isopropyl alcohol and wiped with a waxed cloth. Thus, the part is ready for primer application. The primer which will be applied to the surface was mixed with the hardener and thinner in the amounts recommended in technical data sheet. Subsequently, the application was made with a spray gun. The spray gun has a 1.7 mm nozzle and the application was performed with a pressure of 3-3.5 bar. After the primer application, the part was kept in flash-off time. With this process, the volatile chemicals which are inside of the primer will volatilize before the primer curing and will not make any pinholes on the surface. After the recommended flash-off time, the piece was cured at the recommended temperature and time according to technical data sheet. Thus, the priming process is completed. Before the part surface is prepared for paint application, it has gone through similar steps as in the primer application. The spray gun has a 1.3 mm nozzle and the application was performed with a pressure of 3-3.5 bar. After all the steps were completed, the part was painted, kept in the flash-off time and ovened. In order to test the painted parts, the parts were conditioned for 1 week under room conditions which are 23±2°C and relative humidity of 50±10%. The samples and abbreviations are indicated in Table 1.

Table 1
Abbreviations of samples and their explanations

Abbreviation	Sample's explanations
0% SAFP	Non-treated flax fiber reinforced polyester composite
1% SAFP	1% stearic acid treated non-gelcoated flax fiber reinforced polyester composite
2% SAFP	2% stearic acid treated non-gelcoated flax fiber reinforced polyester composite
3% SAFP	3% stearic acid treated non-gelcoated flax fiber reinforced polyester composite
4% SAFP	4% stearic acid treated non-gelcoated flax fiber reinforced polyester composite
0% SAGFP	Gelcoated and non-treated flax fiber reinforced polyester composite
1% SAGFP	Gelcoated and 1% stearic acid treated flax fiber reinforced polyester composite
2% SAGFP	Gelcoated and 2% stearic acid treated flax fiber reinforced polyester composite
3% SAGFP	Gelcoated and 3% stearic acid treated flax fiber reinforced polyester composite
4% SAGFP	Gelcoated and 4% stearic acid treated flax fiber reinforced polyester composite
0% SAPGFP	Painted, gelcoated and non-treated flax fiber reinforced polyester composite
1% SAPGFP	Painted, gelcoated and 1% stearic acid treated flax fiber reinforced polyester composite
2% SAPGFP	Painted, gelcoated and 2% stearic acid treated flax fiber reinforced polyester composite
3% SAPGFP	Painted, gelcoated and 3% stearic acid treated flax fiber reinforced polyester composite
4% SAPGFP	Painted, gelcoated and 4% stearic acid treated flax fiber reinforced polyester composite

3. Test methods

FT-IR test was performed to control the structure as a result of chemical treatment in the fibers. NICOLET-IS50 branded device was used. The FT-IR method is used to identify test specimens with infrared light and determine their chemical properties. The ISO 2808 standard was used to measure the thickness of the gelcoat, primer and paint applied on the samples. Erichsen Paint Bohrer 518 was used to measure dry layer film thickness (drill 5 for paints and drill 1 for gelcoats). This measurement was realized to check whether the thickness of the gelcoat, primer and paint applied to the samples is within the range of thicknesses recommended in the technical data sheet of the materials. In this way, also total thickness of coating is measured in order to make the right blade selection for cross-cut test. The ISO 62 standard Method-1 method was used for the water absorption test. For this test JSR 13-C branded device was used. Within the scope of this test, the sample dimensions were prepared for 61x61 mm. EN 13523-27 standard was used for the cataplasm test, and the samples were kept in an oven at 60°C for 7 days. Within the scope of this test, the sample dimensions were prepared as 75x150 mm. ISO 4628-2 standard was used to evaluate the blister degree of samples after cataplasm test. The cross-cut test was performed to see the adhesion performance of primer and paint to the composite surface after exposed to humidity for visual assessment. The standard used for this test is the ISO 2409. For cross-cut test TQC SP1690 device was used and Tesa 4657 tape was used to check the adhesion. Before this test, the total paint and primer thickness was measured and the suitable blade was selected. The pull-off test is performed according to ISO 4624 to see the adhesion performance of primer and paint to the composite surface after exposed to humidity for strength assessment. Elcometer 108 device used for this test. The test result is obtained by applying the pulling force to the hydraulic pin which is placed on the painted part.

4. Results and discussion

4.1. FT-IR results

FT-IR analysis was performed before and after the flax fiber was treated with stearic acid. In addition to comparison, stearic acid was also analyzed. Peaks were analyzed between 4000-500 cm^{-1}. The FT-IR results of untreated flax fiber was given in Fig.2a. The peak at 3286.92 cm^{-1} indicates the OH bond in untreated flax fiber. The peak at2896.24 cm^{-1} represents the C-H groups. These peaks belong to the cellulose and hemicellulose substances in the structure of flax fibers. The broadpeak between 1157.94 cm^{-1} and 1103.31 cm^{-1} is attributedto C-O bond. Salem et al. [8], Madhu et al. [15], Zafeiropouloset al. [16], Sathish et al. [17], and Kommula et al. [18] observed these peaks when they examine the natural fibers in FT-IR analysis. Fig.2b displays the FT-IR spectrum of stearic acid. Some distinct peaks were observed at 2953.66 cm^{-1}, 2913.25 cm^{-1}, 2846.90 cm^{-1}. These peaks belong to the CH_2 and CH_3 groups and are found in stearic acid. Li et al. [19] studied stearic acid surface modification of chemicals and observed the same peaks. The broad peak at 1698.01 cm^{-1} is attributed to the carboxylic acid group (C=O).

The FT-IR spectrum of stearic acid treated flax fiber was given in Fig.2c. It is evident that the intensity of the peak around 3286.92 cm^{-1}, associated with hydrogen-bonded O-H stretching, diminishes after the treatment. FT-IR analysis showed that when flax fiber was treated with stearic acid, the spectrum became more similar to the FT-IR spectrum where only stearic acid was measured. This suggests that the flax treated with stearic acid possesses a hydrophobic chain consisting of stearic acid molecules anymore, adding water resistance to the composite [8]. During the treatment process, stearic acid's carboxyl group is expected to interact with the hydroxyl groups present in natural fibers [4]. The FT-IR results are similar to those obtained by Salem et al. [8].

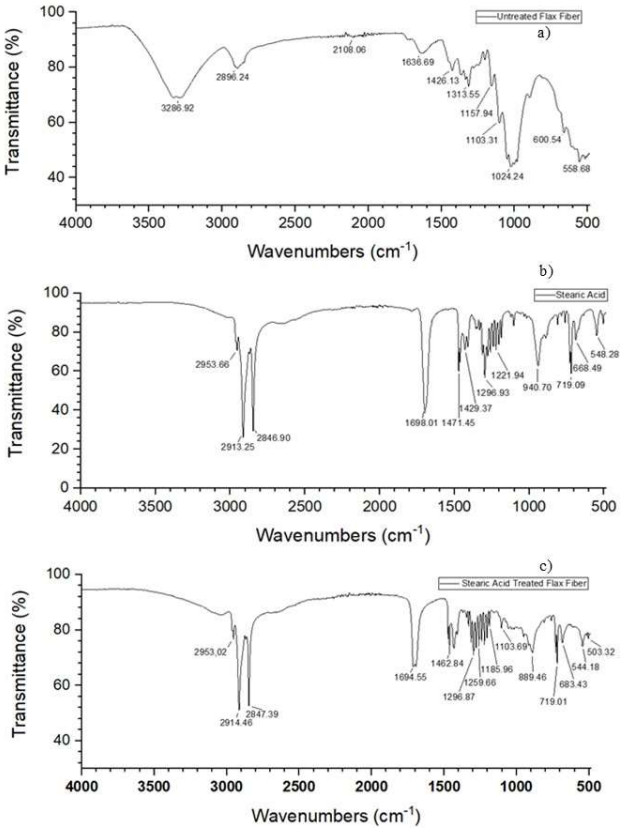

***Fig.2**. FTIR spectra of the untreated flax fiber (a), stearic acid (b) and stearic acid treated flax fiber (c)*

4.2. Water absorption test

The water absorption test results show the amount of water absorbed by the natural fibers after being treated with different percentages of stearic acid for different time intervals. The percentage of stearic acid refers to the amount of stearic acid added to the natural fiber during treatment. The time intervals represent the time for which the treated fibers were exposed to water. The water absorption test samples are given in Fig.3.

Tables 2-4 show 2, 24 and 48 h water absorption test result of non-gelcoated, gelcoated and gelcoated /painted flax fiber reinforced composite which is treated with different ratio of stearic acid. In Table 2, when all the results are evaluated, the water absorption of 3% stearic acid treated flax composite is better than the others. This improvement can be observed for 2 h and 48 h results. If the chemical treatment with stearic acid is more than 3% ratio, the negative effect was seen for this group. The best result of 4% stearic acid treated flax composite is observed for 24 h test. The reason of that is, the test sample dimensions correspond to small area of whole composite panel and due to produce the test panel with hand-lamination process there can be the weight deviations of samples because of being inhomogeneity. In Table 3, when all the results are evaluated, it was observed that the water absorption property of 4% stearic acid treated flax composite is decreasing as the ratio of chemical treatment increased in all samples. As the same like first group of samples, the reason of different result can be that the fact of inhomogeneous structure of composite materials. When the results in Table 3 are compared to Table 2, it was observed that there is positive effect of gelcoat application for water absorption property. The reason of that is, gelcoat covers all the surface of composite and behaviors as barrier so that protect the fibers from the water. In Table 4, when the results are evaluated, it was observed that the water absorption property of 3% stearic acid treated flax composite is decreasing as the ratio of chemical treatment increased in all samples, except the water absorption of 24 h sample. As the same like other two groups of samples, the reason of different result can be that the fact of inhomogeneous structure of composite materials. The gelcoat behaviors as the barrier on the covered surface of composite, due to that the water absorption improvement can be observed. Besides gelcoat, there was more strong barrier behavior observed by using primer and paint. In this way, water penetrate to the panel more difficultly.

Table 2

Water absorption values for non-gelcoated stearic acid treated flax fiber reinforced composites (SAFP)

Non-Gelcoated Percentage of Stearic Acid/Hours	2 h	24 h	48 h
0% SAFP	0.382	0.882	1.242
1% SAFP	0.377	0.819	1.156
2% SAFP	0.334	0.698	1.125
3% SAFP	0.203	0.673	0.905
4% SAFP	0.285	0.665	1.089

Table 3

Water absorption values for gelcoated stearic acid treated flax fiber reinforced composites (SAGFP)

Gelcoated Percentage of Stearic Acid/Hours	2 h	24 h	48 h
0% SAGFP	0.389	0.593	0.854
1% SAGFP	0.332	0.584	0.723
2% SAGFP	0.326	0.522	0.786
3% SAGFP	0.273	0.503	0.674
4% SAGFP	0.221	0.453	0.661

Table 4

Water absorption values for painted, gelcoated and stearic acid treated flax fiber reinforced composites (SAPGFP)

Gelcoated And Painted Percentage of Stearic Acid/Hours	2 h	24 h	48 h
0% SAPGFP	0.255	0.454	0.622
1% SAPGFP	0.167	0.320	0.430
2% SAPGFP	0.152	0.318	0.424
3% SAPGFP	0.104	0.328	0.412
4% SAPGFP	0.073	0.267	0.401

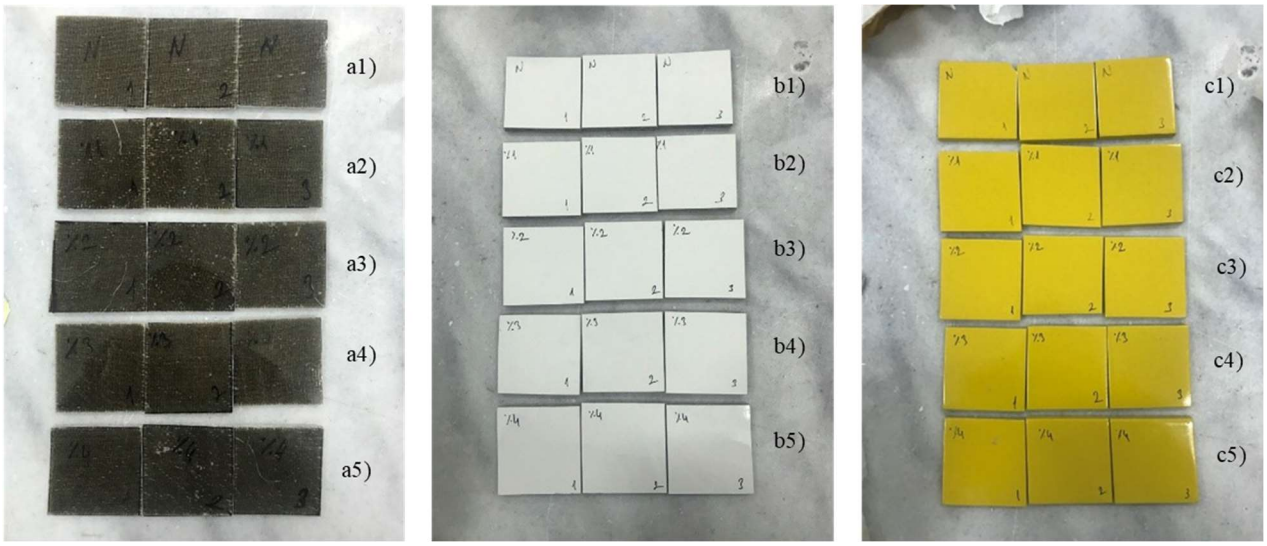

Fig.3. *Water absorption test samples for (a) non-gelcoated composites (a1-0%SAFP, a2-1% SAFP, a3-2% SAFP, a4-3% SAFP, a5-4% SAFP) (b) gelcoated composites (b1-0% SAGFP, b2-1% SAGFP, b3-2% SAGFP, b4-3% SAGFP, b5-4% SAGFP), (c) gelcoated and painted composites (c1-0% SAPGFP, c2-1% SAPGFP, c3-2% SAPGFP, c4-3% SAPGFP, c5-4% SAPGFP).*

From the results, the water absorption is increasing in direct proportion to exposed time to water. At the same exposure time, the water absorption improvement is observed related to increasing stearic acid ratio. It can be seen that as the percentage of stearic acid increases from 0 to 4%, the water absorption of the samples decreases. The reason of that is that stearic acid is a hydrophobic material, which means it repels water and reduces the ability of the composite to absorb water. Overall, the results show that stearic acid treatment can reduce the water absorption of natural fibers. Comparing the three tests of results, it can be seen that the gelcoated and painted fibers generally absorbed the least amount of water compared to the fibers that were just gelcoated or non-gelcoated at all. This shows that the usage of paint and primer improved the water resistance of the fibers. Additionally, it is interesting to note that when the fibers were exposed to water for long term, they absorb more water. The reason of that is, water has more time to penetrate the surface of the fibers and diffuse throughout the material.

Huner [20] investigated the effect of water absorption on the mechanical properties of flax fiber reinforced epoxy composites. Author measured the water absorption properties of the samples produced in his study. As the flax fiber ratio in the composites increased, water absorption properties also increased. When all graphs were examined independently of the flax fiber ratio, it was understood that the water absorption rate increased linearly every hour. Salem et al. [8] stated that water absorption properties improved as the rate increased in chemical treatment with stearic acid. As seen in FT-IR results, when hydrophobicity is added to the structure, a tendency of decreasing water absorption properties is observed. Sreenivasan et al. [11] found a decrease in the water absorption properties of natural fibers chemically treated with stearic acid. As seen in the study of Jain et al. [12], 4% stearic acid treated natural fiber gave the best result in water absorption properties. The water absorption results support the articles. The accuracy of FT-IR measurement can also be confirmed from the water absorption results.

4.3. Dry layer film thickness results

According to technical data sheet values, the primer thickness should be between 70 and 100 µm and the paint thickness should be between 50 and 60 µm. After the paint thickness measurement control, it was seen that the primer thickness is 74 µm and the paint thickness is 59 µm measured values are in range of recommended spec in technical data sheet. Dry layer film thicknesses are given in Fig.4. For gelcoat, 500 µm should have been applied according to the technical data sheet received from the supplier. When the thickness is checked, it is seen that 500 µm gelcoat is applied. Gelcoat thickness is given in Fig.5. Pavelka et al. [21] and Aračić et al. [22] and used the ISO 2808 standard to measure the paint thickness.

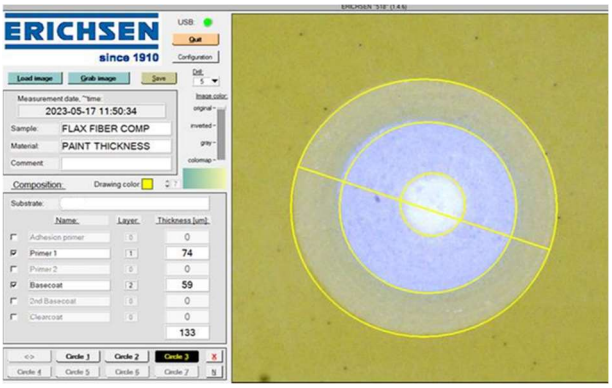

Fig.4. *Dry layer film thickness measurement of painted sample*

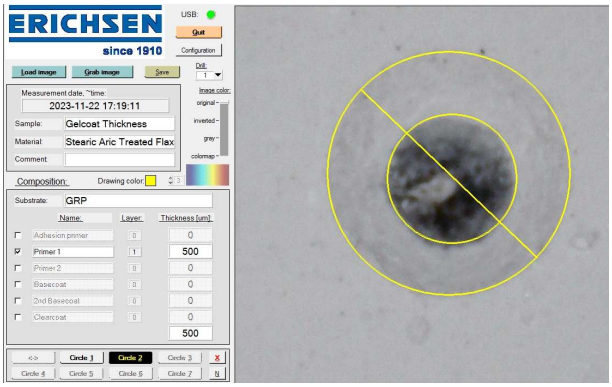

Fig.5. *Gelcoat thickness measurement of the sample.*

4.4. Blister control after cataplasm test

For cataplasm test, the sample was exposed to 60°C temperature for 7 days with wetted cotton. After exposure, it was checked whether blisters were formed on the surface of the painted sample. As a result of the controls, no blisters were found in any of the samples. It has been determined that the product has gained resistance to moisture with the application of primer and paint. Blister and cross-cut test results are given in Table 5.

Table 5
Blister and cross-cut test results

Sample	T(°C)	Blister	Cross-cut after 2 h	Cross-cut after 24 h
0% SAPGFP	60	No blister	GT0	GT0
1% SAPGFP	60	No blister	GT0	GT0
2% SAPGFP	60	No blister	GT0	GT0
3% SAPGFP	60	No blister	GT0	GT0
4% SAPGFP	60	No blister	GT0	GT0

Rosales et al. [23] studied 12 different coils on metal surface. Blister analyses were performed in the study. Garbacz [24] investigated the anti-graffiti properties of paints used in the railway sector. At the same time, the samples were aged in laboratory environment. Blister formation was observed in some samples after ageing. Aračić et al. [22], studied on paint systems to make steels resistant to corrosion. Blister control was performed on the surface of the samples after ageing.

4.5. Cross-cut test

After the paint thickness measurement, blade number 2 was chosen according to the standard for the cross-cut test. This blade was attached to the device and the painted surface of the sample was cut cross sectionally. Tape with 4.6 N/cm strength was attached to the scratched area and the tape was pulled strongly perpendicular to the surface. The surfaces of all samples were checked and no peeling was observed on any painted surface. This test was performed 2 h and 24 h after the end of the cataplasm test. In particular, the results after 2 h usually more critic compare to 24 h, while no peeling was observed in any of the samples. Even after humidity, no decrease in the adhesion performance of the primer and paint was observed.

When the effect of stearic acid treatment is evaluated on cataplasm and cross-cut test results, it was observed that there is no similar effect like water absorption. The results of flax reinforced composite panel with untreated and maximum ratio stearic acid are similar. In this test, it was observed that gelcoat and paint application covered the surface of the composite and provided a reduction in water penetration like similar to water absorption. Based on this barrier behavior, blister was not detected on the sample surface. Cataplasm test result of the samples is given in Fig.6. Pavelka et al. [21] and Aračić et al. [22] performed a cross-cut test after ageing the specimens.

4.6. Pull-off test

Pull-off test samples of before and after cataplasm test are shown in Fig.7. The cataplasm test was performed as aging procedure. The samples are exposed to the humidity under defined temperature and the performance of samples are controlled after testing. The first conclusion that can be obtained from this test is that the application of stearic acid does not have a negative effect on the adhesion of the primer and paint which are applied to the surface. When the samples are examined before aging, this kind of failure can be observed due to fact of not completing the curing degree. After cataplasm test, the samples were aged and, in this way, complete the curing. The curing of paint is better than before cataplasm test, so that failure types can be obtained in optimum results. After cataplasm test, although the strength of 0% SAPGFP sample is less than the others, the failure type is as expected. The reason of this deviation can be the insufficient surface treatment (sanding) before primer and paint application. Surface preparation is important factor for this kind of tests [7]. The reason of the strength value of before cataplasm test is less than after cataplasm test is that the positive affect of aging on curing degree of samples. Mayer et al. [25] and Dmitruk et al. [26] analyzed using this standard in their studies. Pull off test results for before and after cataplasm test are given in Table 6.

Table 6

Pull off test results (before & after cataplasm test)

Sample	Before Cataplasm Test	After Cataplasm Test
0% SAPGFP	3 MPa	4 MPa
1% SAPGFP	7 MPa	5 MPa
2% SAPGFP	5 MPa	5 MPa
3% SAPGFP	5 MPa	6 MPa
4% SAPGFP	6 MPa	7 MPa

Fig.7. *Pull off test results for (a) 0% SAPGFP-before aging, (b) 0% SAPGFP-after aging, (c) 1% SAPGFP-before aging, (d) 1% SAPGFP-after aging, (e) 2% SAPGFP-before aging, (f) 2% SAPGFP-after aging, (g) 3% SAPGFP-before aging, (h) 3% SAPGFP-after aging, (i) 4% SAPGFP-before aging, (j) 4% SAPGFP-after aging*

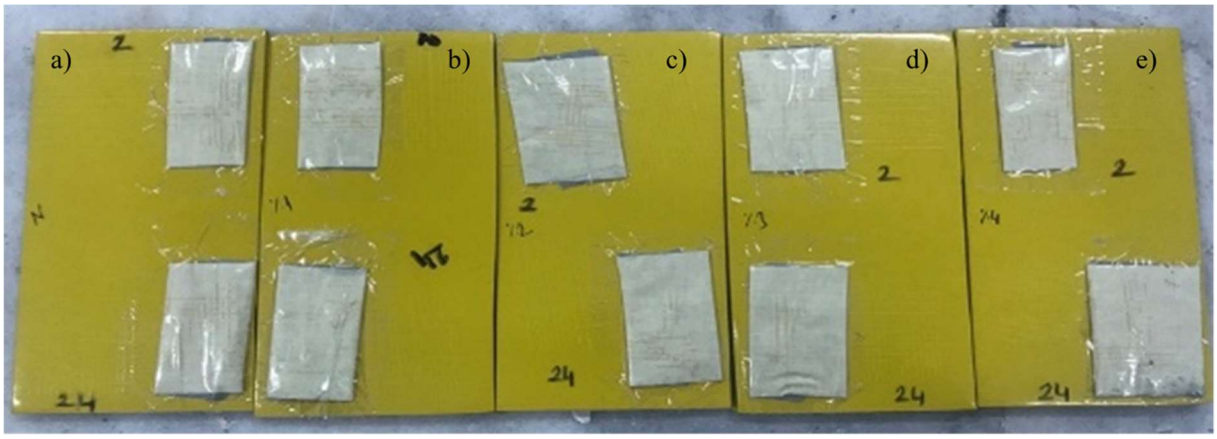

Fig.6. *Cataplasm test for painted samples (a) 0% SAPGFP, (b) 1% SAPGFP, (c) 2% SAPGFP, (d) 3% SAPGFP, (e) 4% SAPGFP.*

5. Conclusion

The flax fiber which is one of the natural fibers that has high water absorption capability. Some chemical treatments are necessary to improve this property. In this study, the flax fiber was treated with different ratios (0, 1, 2, 3 and 4%) of stearic acid. Three different groups were produced. These are; non-gelcoated (FP), gelcoated (GFP) and gelcoated and painted (PGFP). In the FT-IR analysis of untreated flax fiber, a peak at 3286.92 cm^{-1} was observed. This peak disappeared when the flax fiber was treated with stearic acid. However, strong CH_2 and CH_3 bonds in the FT-IR spectrum of stearic acid were also observed in stearic acid treated flax fiber. This confirms that the chemical treatment was successful and the purpose of the reaction was achieved. When the water absorption results were analyzed, it was observed that there was less water absorption in the gelcoat and primer-paint applied groups. This confirmed that gelcoat and primer-paint cover the surface and act as a barrier between water and flax fiber reinforced composite material. As a result of stearic acid treatment at different rates, the groups treated with 4% stearic acid gave the lowest water absorption value. This situation is the same for all 3 groups. As the stearic acid ratio increased, the water absorption tendency of the composite material decreased. Water absorption result confirms the results of FT-IR analysis. The thickness measurement of gelcoat and paint show that the application of both materials was done in range of recommended in technical data sheets. Being inside of the recommended range has positive effect to reduce the possible failures. The cataplasm test is an aging application. As a result of this aging, the appearance of fiber marking on the painted surface on the composite panel were observed. It is normal to see such an effect as a result of aging. When blister control was performed after the cataplasm test, there is no blister was observed in any sample. The adhesion performance of the primer and paint was examined in the cross-cut test after the cataplasm test. Primer and paint did not peel from the surface in any of the samples. This shows that there is no negative effect of aging the sample. The fact that the primer and paint did not peel off the surface after cataplasm may be because of the hydrophobic behavior of stearic acid treated flax fibers. Pull off test results showed that the adhesion of the aged specimens was better. The reason for this may be that the material has not yet completed its curing before aging. Better adhesion may occur in the composite that has completed its curing with cataplasm aging. The results with deviation can be obtained due to insufficient surface treatment, manual human application of gelcoat and paint which can cause higher margin of error. Besides these reasons, the composite structure is inhomogeneous. Hand lamination process performance is mostly depending on the performance of operator. So that the samples which are cutted from composite panel can be inhomogeneous.

Conflicts of interest

The authors declare that they have no conflict of interest.

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References

[1] D. Chandramohan, K. Marimuthu, A Review on Natural Fibers. International Journal of a Research and Reviews in Applied Sciences 8 (2) (2011) 194-206.

[2] M.R. Sanjay, G.R. Arpitha, L.L. Naik, K. Gopalakrishna, B. Yogesha, Applications of Natural Fibers and Its Composites: An Overview. Natural Resources 7 (2016) 108-114.

[3] S. Kalia, B.S. Kaith, I. Kaur, Pretreatments of natural fibers and their application as reinforcing material in polymer composites—A review. Polymer Engineering and Science 49(7) (2009) 1253-1272.

[4] M.Y. Yuhazri, H. Sihombing, Z.A. M. Zaimi, G.C.H. Nilson, A review on gelcoat used in laminated composite structure. International Journal of Research in Engineering and Technology 4 (3) (2015) 49-58.

[5] J.R. Hillman, Hybrid-Composite Beam (HCB) Design and Maintenance Manual. The Missouri Department of Transportation, 2012.

[6] Ç. Paşa, Yıldız Teknik Üniversitesi Fen Bilimleri Enstitüsü, PhD Thesis,20061.

[7] I. Juraga, I. Stojanovic, V. Simunovic, M. Nikolic, shop primers and their influence on welding– shipbuilding Review. Proceeding of the 4th International scientific-professional conference cost effective application of welding processes and welding related techniques in manufacturing of construction and products (2007) 107-115.

[8] I.A.S. Salem, A.R. Rozyanty, B.O. Betar, T. Adam, M. Mohammed, A.M. Mohammed, Study of the effect of surface treatment of kenaf fiber on chemical structure and water absorption of kenaf filled unsaturated polyester composite. Journal of Physics: Conference Series908 (2017) 012001.

[9] M.M. Kabir, H. Wang, K.T. Lau, F. Cardona, Chemical Treatments of Plant-Based Natural Fibre Reinforced Composites: An Overview. Composites Part B: Engineering43 (2012) 2883-2892.

[10] J.A. Halip, L.S. Hua, Z. Ashaari, P.M. Tahir, L.W. Chen, M.K.A. Uyup, Effect of treatment on water absorption behavior of natural fiber–Reinforced polymer composites, In; M. Jawaid, M. Thariq, N. Saba (Eds), Mechanical and Physical Testing of Biocomposites, Fibre-Reinforced Composites and Hybrid Composites, Woodhead Publishing,2019 pp.141-156.

[11] V.S. Sreenivasan, D. Ravindran, V. Manikandan, R. Narayanasamy, Influence of fibre treatments on mechanical properties of short Sansevieria cylindrica/polyester composites. Materials and Design 37 (2012) 111–121.

[12] D. Jain, H. Kaur, T.K. Bera, R. Jain. Comparison of Different Hydrophobic Treatments for the Durability Improvement of Palmyra Natural Fiber Composites under Hydrothermal Ageing Environments. Journal of Natural fibers 17(11) (2020) 1668-1682.

[13] P.I. Dolez, M.A. Arfaoui, M. Dube, E. David, Hydrophobic treatments for natural fibers based on metal oxide nanoparticles and fatty acids. Procedia Engineering 200 (2017) 81-88.

[14] N. Kiattipanich, N. Kreua-ongarjnukool, T. Pongpayoon and C. Phalakornkule, Properties of polypropylene composites reinforced with stearic acid treated sugarcane fiber. Journal of Polymer Engineering 27 (6-7) (2007) 411-428.

[15] P. Madhu, M.R. Sanjay, M. Jawaid, S. Siengchin, A. Khan, C.I. Pruncu, A new study on effect of various chemical treatments on Agave Americana fiber for composite reinforcement: physico-chemical, thermal, mechanical and morphological properties. Polymer Testing 85 (2020) 106437.

[16] N.E. Zafeiropoulos, P.E. Vickers, C.A. Baillie, J. F. Watts,An experimental investigation of modified and unmodified flax fibres with XPS, ToF-SIMS and ATR-FTIR. Journal of Materials Science 38 (2003) 3903-3914.

[17] S. Sathish, K. Kumaresan, L. Prabhu, S. Gokulkumar, Experimental investigation of mechanical and FTIR analysis of flax fiber/epoxy composites incorporating SiC, Al_2O_3 and graphite. Romanian Journal of Materials 48 (4) (2018) 476-482.

[18] V.P. Kommula, K. O. Reddy, M. Shukla, T. Marwala, E.V.S. Reddy, A.V. Rajulu, Extraction, modification, and characterization of natural ligno-cellulosic fiber strands from napier grass. International Journal of Polymer Analysis and Characterization 21 (1) (2016) 18-28.

[19] Y. Li., W. Weng, Surface modification of hydroxyapatite by stearic acid: characterization and in vitro behaviors. Journal of Materials Science: Materials in Medicine19 (2008) 19-25.

[20] U. Huner, Effect of water absorption on the mechanical properties of flax fiber reinforced epoxy composites. Advances in Science and Technology Research Journal 9 (26) (2015) 1-6.

[21] R. Pavelka, M. Brozek, Progressive method of observation and assessment during corrosion tests with artificial atmosphere. Proceeding of the 13th International Scientific Conference on Engineering for Rural Development (2014) pp. 181-186.

[22] S. Aračić, D. Krumes, T. Matičević, Research into the possibility of using the new generation paint systems for the anticorrosion protection of steel structures. Technical Gazette 16 (4) (2009) 75-79.

[23] B.M. Rosales, A.R.D. Sarli, O. Rincon, A. Rincon, C.I. Elsner, B. Marchisio, An Evaluation of Coil Coating Formulations in Marine Environments. Progress in Organic Coatings 50 (2004) 105-114.

[24] M. Garbacz, Influence of Laboratory Ageing on the Preservation of Anti-graffiti Properties for Paint Systems Used in Rolling Stock. Problemy Kolejnictwa 195 (2022) 97-116.

[25] P. Mayer, A. Dmitruk, N. Leja, E. Pakiet, Pull-off strength of fibre-reinforced composite polymer coatings on steel substrate. The Journal of Adhesion 98(3) (2020) 286-304.

[26] A. Dmitruk, P. Mayer, J. Pach, Pull-off strength of thermoplastic fiber-reinforced composite coatings. Journal of Adhesion Science and Technology 32 (9) (2018) 997-1006.