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Areca Catechu Husk Fibers and Polypropylene Blended Nonwovens for Medical Textiles.

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Abstract: Areca Catechu husk fibers is an agri-waste fiber. These husk fibers from the nut shell have limited applications. This fiber has been given alkali pretreatment in an autoclave and delignified using enzymes in order to improve the absorbency of the fibers. Polypropylene fibers have been blended with the prepared arecanut fibers to render the required thermo plasticity for the fusing technique, used for nonwoven preparation. The blended fibers have been made into a web using a mini carder machine, further to which the webs have been fused in a fusing machine. The fusing process has been optimized through trial and error method by altering the fusing machine variables of temperature, pressure and time. The prepared webs have been tested for various performance properties and are compared with the properties of the commercially available polypropylene sheets. The tests done on the prepared fused nonwoven made of areca catechu / polypropylene fibers have proved that the aforesaid nonwovens are suitable for medical textiles especially for cover stocks of sanitary napkins. Combining bio-based materials with PP fibers to develop an effective product having many applications is a better way to follow sustainability.

Keywords : Natural fibers, Areca catechu husk fibers, Arecanut husk fibers, polypropylene, Lacasse enzyme, Delignification, fusing, nonwovens, medical textiles, sanitary napkins, cover stock.

Introduction

The increasing awareness about hygienic lifestyle among the consumers, increases people's expectation on textiles, for rendering this hygienic functionality. [1] A lot of research in the textile and the apparel fields try to focus on giving this comfort to the wearer by adapting various new technologies leading to innovative fibers, fabrics, finishes and new apparel products. Many commercial products available in the market are synthetic ones. But in order to meet the global standards of eco-friendliness and sustainability, market is now penetrated by safer natural products which have the capability of giving multi functionalities to our textiles.[2] [3] & [4]. A product is said to be green based on the intrinsic characteristics of the product, using recycled raw materials [5]. Ecological concerns have resulted in a resumed interest in renewable resources-based products. It is the reason why material components such as natural fibers and biodegradable polymers can be considered as environmentally safe – alternative. [6]. All natural fibers have low density, less abrasive to processing equipments, biodegradable and economically viable [7]

Wastes and by-products from agro-industrial processes such as coconut shells, rice husks, sugarcane bagasse, corncob and corn stover among many others, are abundantly produced in the world daily and have modest if any applications. These wastes and by-products are rich sources of cellulose and hemicelluloses wrapped up in lignin, which is an inert polymer that protects the plant [8]. It belongs to the species Areca Catechu Linnaeus under the family Palmacea. Among all the natural fibers Areca nut fibers, a type of nut shell fibers, are more promising because it is inexpensive, abundantly available and very high potential perennial crop but have limited applications [9]. Areca nut fibers are hypoallergenic and have inherent antimicrobial characteristics. The polyphenols of ripe arecanut contain predominantly polymerized leucocyanidins, besides minor amounts of (+)-catechin, leucopelargonidin and leucocyanidin. The polyphenols of arecanut are mainly flavonoids, and their concentration decreases with the maturity of the nut [10].

Not much of applications are available for these areca husk fibers. However, a part of these fibers are used as fuel and most of them are left as a waste in landfills and are difficult to manage. Improvements done to the areca nut fibers can find new applications for these fibers. Alkali treatment improves fiber strength, fiber matrix adhesion and the performance of the natural fiber composites.[11, 12] The husk of the fiber consists about 60% to 80% of the total weight of fresh fruit. The average filament length is around 4 cms. which is too short compared to other bio-fibers. Mainly 2 types of fibers are present – one very coarse and the other very fine. The coarser ones are ten times coarser than jute [9]. These fibers adjoining the inner layers are irregularly lignified group of cells called hard fibers. The portions of the middle layer below the outermost layer are soft fibers, which are very similar to the jute fibers [13]. Vegetable fibres are generally composed of three structural polymers (the polysaccharides cellulose, and hemicelluloses and the aromatic polymer lignin) as well as by some minor non-structural components (i.e. proteins, extractives, minerals) [13 14]. The arecanut husk fiber also is composed of cellulose with varying proportions of hemicellulose (35–64.8%), lignin (13.0–26.0%), pectin and protopectin. [15]

The cell walls of plant cells consist of cellulose fibrils embedded within a matrix of lignin and hemicellulosic polysaccharides [16]. Lignin is the main constituent of arecanut fiber, responsible for hardening of plant cell wall and the reason for the fiber stiffness. [10 9]. So these hemicellulose and lignin has to be removed for improving the performance of the fibers. Delignification is the process of removal of lignin and is used to improve the absorbency of the fibers[17]. This lignin removal can be done through various enzymatic methods which are commonly known as enzymatic delignification. Bio-delignification can be produced by the action of lignin peroxidase (LiP), manganese peroxidase (MnP), laccase, and versatile peroxidase (VP)[9 8].

The main objective of this study is to develop a nonwoven material from delignified arecanut husk fibers blended with polypropylene fibers. This fiber has been selected and prepared to various stages to improve its absorbency. This blending of natural and synthetic fibers will help in reducing the content of synthetic fibers in nonwovens thereby also reduces the land fill considerably. New applications of agri waste fibers like arecanut husk fibers will focus on recycling the agriwaste and facilitate sustainability.

Experimental

Materials:

Areca Catechu:

Arecanut fibers are extracted from its matured and dried fruit. It is composed of both thick fibres and fine fibres. The finer fibers were extracted from the nut and used for further processing.

Polypropylene fibers:

Polypropylene fibers have been selected to blend with the prepared areca nut fibers to render the required thermo plasticity for the fusing technique of preparing nonwovens

Sapindus Mukorossi:

Areca catechu fibers were cleaned with Sapindus Mukorossi, an organic cleaning agent, in order to remove natural wax content and impurities.

Chemicals:

Sodium hydroxide has been used for pretreating the Areca Catechu fibers. Later these fibers were delignified using laccase enzyme (Bactosal LAC powder) which was supplied by Clariant Chemicals Ltd, India for the removal of lignin.

Methods

Cleaning of Areca Catechu fibers with Sapindus Mukorossi (puchakai)

For cleaning organic cleaning agents called **Sapindus mukorossi** (puchakai) were used. Since ancient times, Sapindus mukorossi has been used as a detergent for shawls, silks and as a hair shampoo due to its rich natural lather. The fruit of Sapindus mukorossi was utilised by Indian jewellers for restoring the brightness of tarnished ornaments made of gold, silver and other precious metals.[18] 10 % concentration of **Sapindus mukorossi** solution was prepared to treat Areca Catechu fibers with a M:L ratio of 1:10 till the boiling point.

Delignification of Areca Catechu fibers:

Pre-treatment of Areca Catechu fibers by alkali cooking:

Alkali treatment of natural fibres is used to produce high-quality fibres by transferring crystallinity from cellulose I into cellulose II. Alkali treatment also removes lignin and hemicelluloses. [19,20] Sodium hydroxide (NaOH) aids the greatest in degradation of lignin when compared to other alkalis, such as sodium carbonate, ammonium hydroxide, calcium hydroxide and hydrogen peroxide [21] & [22]. So in this study the NaOH pre-treatments were given for arecanut fiber in order to reduce the hemicellulose and lignin content. Alkali pre-treatments at lower temperatures give coarser fibers but higher temperatures can give finer fibers. So in this study higher temperature was selected [23]. Areca Catechu fibers were cooked with NaOH alkali in a autoclave, at a pH of 12 with temperature and pressure maintained in a autoclave at 120^{o} C and 13 PSI pressure. [24]. Cooking time was varied by accounting the number of whistles in an autoclave.

Delignification

Delignification is the removal of the structural polymer lignin from plant tissue. This Lignin reduces the absorbency of the fiber and is responsible for the crystallinity of the fibers. So it has to be removed in order to increase the absorbency of the fiber.

After the pretreatment with alkali cooking the Areca Catechu fibers are processed with the lacasse enzyme. The bath is set with M:L ratio of 1:10, pH 5-5.5, and temperature maintained at 65ºC and required concentrations of Bactosol (lacasse enzyme) are added and the fibers are treated for 30 minutes at 70ºC. Later fibers are rinsed in plain water and dried in the oven at 135ºC temperature.

2 different ways of preparing Areca Catechu fibers were followed by varying the alkali % and cooking time during the alkali cooking of the fibers. Later the alkali cooked fibers were delignified with 2 different laccasse enzyme concentrations. Table 1 clearly shows the variables of the prepared 2 fibers namely DAF and DBF

Table 1: Prepared Fibers to Develop the Fused Nonwovens

Fiber sample	**Alkali cooking**		**Delignification**
	Alkali (%)	**Cooking time in (No. of whistles)**	**Enzyme concentration (%)**
DAF	5	35	6
DBF	6	40	6

Optimizing the process parameters of the fusing process:

In order to find a non woven technique, to prepare the cover stock from the prepared Areca Catechu fibers, fusing method has been selected as it is a cost effective method and could be adhered for smaller quantities of fibers. The processed fibers were blended with polypropylene fibers to achieve the thermo plasticity required for the fusing technique as given in the table 2. PP-F nonwovens comprise of 100% PP fibers and PP-SB was the commercially available spun bond nonwoven sheet comprising 100% PP fibers, which is generally used for most of the medical textiles. The fibers were prepared into webs in a mini carder machine. These webs were used to prepare nonwoven sheets using the fusing technique.

Table 2: Nonwoven Samples Prepared through Fusing Process

S.No	Sample name	Description of the fiber composition
1.	DAF-25	Arecanut : PP -25/75
2.	DAF-50	Arecanut : PP -50/50
3.	DAF-75	Arecanut : PP -75/25
4.	DBF-25	Arecanut : PP -25/75
5.	DBF-50	Arecanut : PP -50/50
6.	DBF-75	Arecanut : PP -75/25
7.	PP-F	PP – 100%
8.	PP-SB	PP – 100%

By using trial and error method the parameters for preparing the fused nonwovens have been selected. During the fusing process the fibers will melt and stick to the surface of the fusing belts. To avoid this, food grade aluminum foil is wrapped around the web and then passed between the rollers for fusing. During fusing the web gets elongated along the width due to melting of the fibers. Due to this the centre portion was thinner and the edges of the web had a thick layer. So to find an alternative for this problem, instead of wrapping the web with the aluminum foil, the foil is just placed on the top and bottom of the web and this arrangement is placed between sheets of news papers and then passed between the rollers for fusing. This prepared even non woven sheets. By changing the variables of the fusing machine like temperature, pressure and speed, various trials were done and samples were prepared as indicated in the Table 3.

Table 3: Variables used to Optimize the Fusing Process

Various trials taken	Variables for preparing the various trials of fused nonwovens					
	Temperature (^{o} C)		Pressure (bar)		Speed m/mins	
	100	120	3	4	5	10
Trial 1	X		X		X	
Trial 2	X		X			X
Trial 3	X			X	X	
Trial 4	X			X		X
Trial 5		X	X		X	
Trial 6		X	X			X
Trial 7		X		X	X	
Trial 8		X		X		X

Dimensional and comfort properties of prepare fused nonwovens:

The prepared varieties of fused nonwovens were tested for their dimensional and performance properties using the standard test methods as given below in the Table 5

Table 4: Descriptions of Cover Stock Samples Prepared through Fusing Process

S.No	Experiment	Standard
1	GSM	ASTM D 2646
2	Air permeability	Airtronic permetester ASTM D6476
3	Water vapour permeability	Water vapour permetester ASTM E9680
4	Tearing strength of the cover stock	Instron ASTM D2261
5.	Wicking	Vertical wicking test ASTM D1777
6.	Thickness	Thickness tester ISO 3616

Results and Discussion

Optimizing the process parameters of the fusing process:

The following were observed at the end of the trials. Higher temperatures, higher pressures and lower speed in the fusing process led to increase in the melting of the polypropylene fibers and formed stiff and film like non woven sheets. The melting had to just help in fiber adherence but in this case the fibers melted more than required and had formed film like sheets which will affect the wickability of the nonwovens. Lower temperature, lower pressure and higher speed gave better fused nonwovens. The trial 2 sample gave a better fused nonwoven sample with the desirable results. The parameters for preparing the fused nonwoven sample was selected as Temperature -100 ^{o}C, Pressure - 3 bar and Speed of 10 mts/mins. This trial enabled the fusing process to control the melting of the polypropylene fibers and create fibrous nonwovens.

Dimensional Characteristics of the Prepared Nonwoven Samples:

The Figure 4 depicts the weight of the fused nonwoven samples in grams per square meter. PP-SB and PP-F are 100% polypropylene (PP) fibers and they are least in weight when compared to the blended fibers. When comparing between PS-SB sample and PP-F, they differ in the manufacturing method, one being spun bond and the other being fused. Still the difference in the GSM of both samples is negligible.

When observing the blended samples the weight achieved ranged between 28.32 grams/square meter and 34.52 grams/square meter. All the samples made of DAF fibers have weighed lesser than the samples made of DAB fiber. This is due to the fact that the alkali pre treatment and delignification process has removed more quantity of hemicellulose and lignin in the case of DAF than when compared to DBF fibers.

When comparing within DAF and DBF fiber samples, samples with lesser quantity of Areca Catechu fibers have weighed less. The more the composition of Areca Catechu, the more the samples weigh. Fused nonwoven samples with 75% arecanut fibres weigh higher. This is due to the presence of more Areca Catechu fibers which are heavier than the PP fibers.

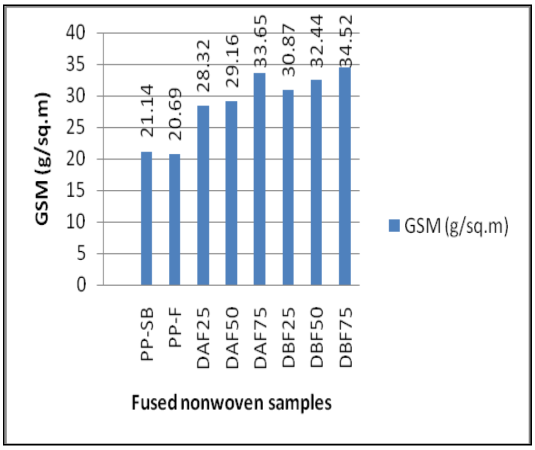


Fig.4: GSM of nonwoven samples

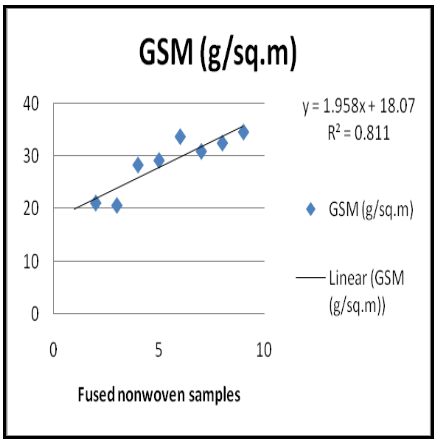
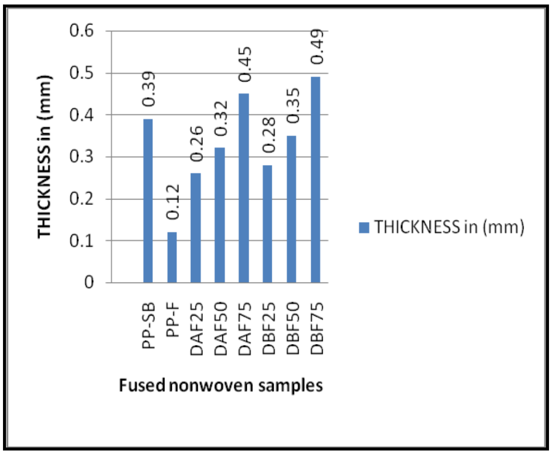


Fig.5: Scatter plots for GSM of nonwoven samples

The Regression Coefficient values (R^2) value 0.811, of linear regression curve in Fig 5, proves that there is a linear relationship among all the weights achieved in all the fused nonwoven samples. Increase in Areca Catechu fibers increase the weight of the nonwoven samples. The Regression equation is achieved as $y = 1.958x + 18.07$



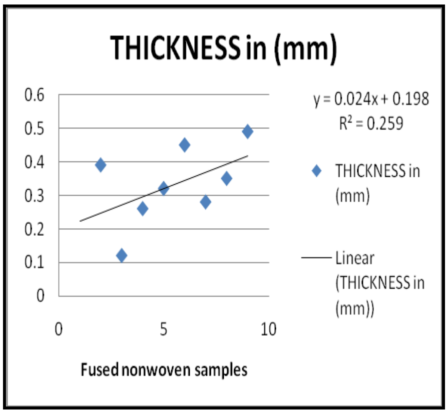


Fig.6: Thickness of nonwoven samples **Fig.7: Scatter plots for Thickness of nonwoven samples**

Fig.6 presents the thickness of the prepared nonwoven samples. PP-SB is 0.4 mm which is higher than that of PP-F sample which is only 0.12mm. PP-F has recorded the least thickness among all the samples. This is due to the difference in the manufacturing method of both the samples. During the fusing process there is an even application of temperature and pressure on the web due to which more fibers are subjected to melting. But in the case of spun-bond method of nonwoven preparation, binding points which integrate the fibers, are present only at patterned intervals where the melting happens. Due to this the PP-SB have more freedom and therefore have more air space. Due to this reason the PP-F sample is lesser in thickness.

All the blended samples have recorded thickness in the range of 0.3mm-0.5mm which are much thicker than the PP-F samples but are very much similar to the thickness of PP-SB nonwovens which is almost 0.4 mm thickness. This is due to the thickness of the Areca catechu natural fibers, whereas the PP fibers are much thinner. So the application of Areca Catechu fibers with PP fibers and fusing them to form a nonwoven sheet has helped in preparing a nonwoven which is similar to the thickness of PP-SB commercially available polypropylene non-woven. As the natural fibers are not thermoplastic, they do not melt and will help to maintain the air spaces as in spun bond method of nonwoven manufacturing

The Regression Coefficient values (R^2) value 0.259, of linear regression curve in Fig 7, proves that there is no linear relationship among the thickness of various prepared fused samples. This unevenness in the thickness of the nonwovens is due to the unevenness of the Areca Catechu fibers, which is the characteristic nature of natural fibers. The Regression equation is achieved as $y = 0.024x + 0.198$

Performance Characteristics of the Prepared Nonwoven Samples:

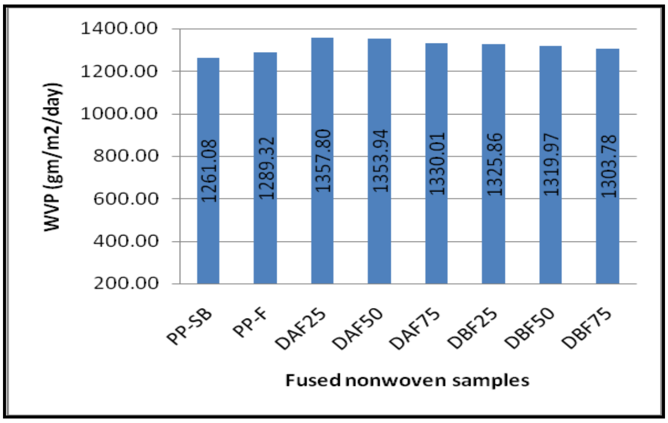


Fig.8: Results for Water Vapor Permeability of Fused Nonwoven Samples

Figure 8 shows the results for water vapor permeability (WVP) of the fused nonwovens. The highest WVP has been recorded by DAF-25 sample with 1357.80 gm/m^2/day. The least WVP has been recorded by PP-SB sample with 1261.08 gm/m^2/day. All the fused samples have recorded higher results for water vapor permeability than the PP-SB sample ranging from 1357.80 to 1303.78 gm/m^2/day. This increase in the WVP is due to the higher GSM of these samples when compared to the PP-SB sample and due to the presence of the natural fiber whose absorbency also has contributed to the WVP. DAF fibers have better WVP results rather than DBF fibers proving better wickability of the DAF fibers due to chemical treatments.

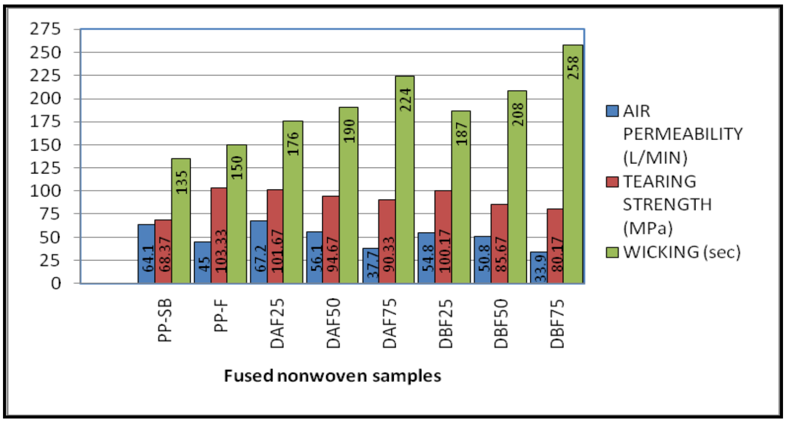


Fig.9: Results for Air Permeability, Tearing Strength and Wicking of Fused Nonwoven Samples

Figure 9 shows the results for Air permeability, tearing strength and wicking ability of the fused nonwovens samples. The highest Air permeability has been recorded by DAF-25 sample with 67.2 L/Min. The least Air permeability has been recorded by PP-F sample with 45 L/Min. All the fused samples have recorded Air permeability ranging from 67.2 to 33.9 L/Min. PP-SB have more thickness but have bonding points at regular intervals which gives more freedom to the fibers and allow more air to pass through but PP-F does not allow so much air to pass. In the case of blended samples, more the presence of natural fibers, there is less air permeability. This is due to the fact that PP fibers are finer than the Areca Catechu fibers.

When comparing between the DAF and DBF samples, DAF samples have better air permeability than the DBF fibers and this is substantiated by the lesser GSM and lesser thickness of the DAF fibers. This again is a proof for the higher loss of portions of the DAF fiber due to delignification undergone by the fibers through alkali pre-treatment and Laccasse treatment of the fibers.

When analyzing the tearing strength of the prepared samples, all the fused samples have shown higher strength ranging between 103.33 MPa to 80.17 MPa, when compared to the PP-SB with 68.37 MPa, which is a spun bonded nonwoven. This is due to the reason that spun bond has less number of binding points, where fibers are melted. But fused samples have more points where fibers are melted, which enhance the tearing strength of the fused samples. When comparing between the blended fibers, more the presence of the natural fibers, there is a decrease in the tearing strength of the fused samples. When comparing between the DAF and DBF fibers, DAF fibers have shown higher tearing strength.

When comparing the wicking of all the samples, PP-SB has the least time of 135 seconds followed by PP-F with 150 seconds. All the fused samples have recorded higher time for wicking than both the 100% PP fibers. The wicking time has increased with increase in the Areca Catechu fiber content ranging from 176 to 258 seconds. Though the absorbency of the Areca catechu has improved due to the chemical processing, this has not helped in improving the wicking of the natural fibers equivalent to perform like the PP fibers. This is viewed as a limitation of the fused samples. But DAF-25 sample is much closer to the wicking of the PP-SB sample.

Conclusion

- The parameters for preparing the PP and Areca Catechu /PP blended fused nonwoven samples was selected as Temperature -100 ^{o}C, Pressure - 3 bar and Speed of 10 mts/mins. The sample with the mentioned parameters enabled the fusing process to control the melting of the polypropylene fibers and create fibrous nonwovens.
- Though PS-SB sample and PP-F differ in the manufacturing method, one being spun bond and the other being fused, still the difference in the weight of both samples is negligible. All the samples made of DAF

fibers weighed lesser than the samples made of DAB fiber which proves the degradation of the fibers as a result of the delignification process by lacasse. The more the composition of Areca Catechu, the more the samples weigh.

- The Regression Coefficient values (R^2) 0.811 of the weights of the prepared fused samples, proves that there is a linear relationship among all the weights achieved in all the fused nonwoven samples. Increase in Areca Catechu fibers increase the weight of the nonwoven samples. The Regression equation is achieved as $y = 1.958x + 18.07$
- PP-F has recorded the least thickness among all the samples. During the fusing process there is an even application of temperature and pressure on the web due to which more fibers are subjected to melting. Due to this reason the PP-F sample is lesser in thickness.
- All the blended samples have recorded thickness very much similar to the thickness of PP-SB nonwovens. This is due to the thickness of the Areca catechu natural fibers, whereas the PP fibers are much thinner. So the application of Areca Catechu fibers with PP fibers and fusing them to form a nonwoven sheet has helped in preparing a nonwoven which is similar to the thickness of PP-SB commercially available polypropylene non-woven. As the natural fibers are not thermoplastic, they do not melt and will help to maintain the air spaces as in spun bond method of nonwoven manufacturing
- The Regression Coefficient value (R^2) 0.259 of the thickness of all the prepared nonwoven samples proves that there is no linear relationship among the thickness of various prepared fused samples. This unevenness in the thickness of the nonwovens is due to the unevenness of the Areca Catechu fibers, which is the characteristic nature of all natural fibers. The Regression equation is achieved as $y = 0.024x + 0.198$
- All the fused samples have recorded higher results for water vapor permeability than the PP-SB sample. This increase in the WVP is due to the higher GSM of these samples when compared to the PP-SB sample and due to the presence of the natural fiber whose absorbency also has contributed to the WVP. DAF fibers have better WVP results rather than DBF fibers proving better wickability of the DAF fibers due to chemical treatments.
- PP-SB have more thickness but have binding points at regular intervals which gives more freedom to the fibers and allow more air to pass through but PP-F does not allow so much air to pass. In the case of blended samples, more the presence of natural fibers, there is less air permeability. This is due to the fact that PP fibers are finer than the Areca Catechu fibers.
- DAF samples have better air permeability than the DBF fibers. This again is a proof for the higher loss of portions of the DAF fiber due to delignification undergone by the fibers through alkali pre-treatment and Laccasse treatment of the fibers.
- All the fused samples have shown higher strength than PP-SB which is a spun bonded nonwoven. This is due to the reason that spun bond has less number of binding points, where fibers are melted. But fused samples have more points where fibers are melted, which enhance the tearing strength of the fused samples. When comparing between the blended fibers, more the presence of the natural fibers, there is a decrease in the tearing strength of the fused samples. When comparing between the DAF and DBF fibers, DAF fibers have shown higher tearing strength.
- All the fused samples have recorded higher time for wicking than both the 100% PP fibers. The wicking time has increased with increase in the Areca Catechu fiber content. Though the absorbency of the Areca catechu has improved due to the chemical processing, this has not helped in improving the wicking of the natural fibers equivalent to perform like the PP fibers. This is viewed as a limitation of the fused samples. But DAF-25 sample is much closer to the wicking of the PP-SB sample.
- In both the dimensional and performance properties, DAF fibers are better than the DBF fibers. Among the 3 DAF fused nonwoven samples DAF-25 has favorable characteristics and are very close to the PP-SB commercial nonwoven sample. This fused nonwoven sample could be used for various applications similar to the PP-SB sample like cover stock for absorbent hygiene products, aprons etc.

This study on preparing fused nonwoven samples from PP and Areca Catechu blended fibers has introduced a new functionality to the agri-waste Areca Catechu husk fibers. This study has paved way to sustainability as the waste has been recycled and has been processed in eco-friendly methods. Combining bio-based materials with PP fibers to develop an effective product having many applications is a better way to follow sustainability [25]

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