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Appendices

Appendix Ia

Ethical Clearance Certificate

INSTITUTIONAL HUMAN ETHICS COMMITTEE

Avinashilingam
Institute for Home Science and Higher Education for Women
University
(Estd. u/s 3 of UGC Act 1956)

Chairman
Dr. S. Ramalingam
Principal, PSG Institute
of Medical Sciences
& Research, Coimbatore

Member Secretary
Dr.S.Uma Mageshwari
Associate Professor,
Department of Food Service
Management & Dietetics

Members
Dr. S. Kowsalya
Dr.P.R.Padma
Mr. K.Arulmoli (Legal Expert)
Dr. N.S. Rohini
Dr.A. Saraswathy
Mrs. V. Mangayarkarasi
Dr.Subhashini K. Sripathi
Mrs. S. Radha Devi
Dr.G.Victoria Naomi
Dr. Judith Justin
Dr.AnithaSubash

19th March 2018

To
Ms. S. Thamarai Selvi
Department of Textiles and Clothing
Avinashilingam Institute for Home Science and
Higher Education for Women
Coimbatore – 641 043

Dear Thamarai Selvi,

Ref: Your proposal No. IHEC/17-18/TC/04 "Fabrication of Natural Fibre for Technical Textile Applications" submitted for approval of the IHEC on 14th December.

The Institutional Human Ethics Committee of our University hereby grants approval to your research proposal No. IHEC/17-18/TC/04 "Fabrication of Natural Fibre for Technical Textile Applications" submitted by you. The Approval number for the same is AUW/IHEC/ TC -17-18/XPD/04.

We wish you all the best in your research endeavours.

Regards,

S. Uma Mageshwari
Dr.S.Uma Mageshwari
Member Secretary

Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore ★ Institutional Human Ethics Committee

Appendix Ib

QUESTIONNAIRE TO ELICIT INFORMATION ON USE OF MATERIALS IN BUILDING CONSTRUCTION AND POTENTIALITY OF NATURAL FIBRES FOR VARIOUS CIVIL APPLICATIONS

1. Name:
2. Gender:
3. Qualification:
4. Designation:
5. Residential Address:

Employment Details

6. **Are you employed?**

   ☐ Yes ☐ No

7. **If Yes, What is the type of company where you are employed?**

7a. Government : ☐

Name and address of the company

7b. Private ☐

Name and address of the company

7c. What is your designation in the company?

Super visor -

Director -

Quality controller -

Managing Director -

Material Assessor -

Meson -

Draftsman -

Any other -

8. What types of job do you under take?

S.No	Jobs Undertaken	Always	Some times	Not at all
1	Excavation work			
2	Demolishing			
3	Site clearing			
4	Basement work			
5	Water proofing			
6	**Flooring**			
a	Plastering			
b	Marble and granite cutting			
c	Marble and granite laying			
d	Pavement / floor laying			
e	Tiles laying			
7	**Roofing**			
a	Concrete work			
b	Any other			
8	**Ceiling**			
a	Interior false roofing			
b	Any other			
9	**Carpentry work**			
a	Doors			
b	Windows			
c	Modular works			
d	Furnishings			
e	Partition jobs			

f	Any other			
10	Road works and Highways			
11	**Painting works**			
a	White washing			
b	Distempering			

9. What type of materials do you suggest/use in your construction for flooring?

S.No	Materials	Very Rarely	Rarely	Moderately	Often	Very Often
	Flooring Materials					
1.	Asphalt					
2.	Bamboo					
3.	Bricks					
4.	Carpet tiles					
5.	Cement					
6.	Ceramics					
7.	Cork					
8.	Composites					
9.	Earthen					
10.	Epoxy					
11.	Flag stone					
12.	Glass Tiles					
13.	Granite					
14.	Laminates					
15.	Leather					
16.	Lime stone					

17.	Linoleum					
18.	Marble					
19.	Mud/ Murram					
20.	PET					
21.	Plastic (PVC)					
22.	Rubber					
23.	Tiles					
24.	Vinyl					
25.	Wood					
26.	Wool					
27.	Any other					

10. What type of materials do you suggest/use in your construction field for roofing?

S.No	Materials	Very Rarely	Rarely	Moderately	Often	Very Often
	Roofing Materials					
1	Asbestos Shingle					
2	Asphalt Shingle					
3	Bamboo					
4	Cement /Concrete					
5	Ceramic tile					
6	Clay tiles					
7	Composites					
8	EPDM (Rubber)					
9	Lime stone					
10	Membrane					
11	Metal sheets					
12	Slate Shingles					
13	Solar Shingle					
14	Stone Slab					
15	Thatch					
16	Turbines					

17	White cement					
18	Wood Shingle					
19	Any other					

11. What type of materials do you use in your construction for Wall covering?

S.No	Materials	Very Rarely	Rarely	Moderately	Often	Very Often
Wall covering (Exterior) Materials						
1	Bricks					
2	Metal Cladding					
3	Mortar					
4	Mud					
5	Plastic					
6	Stone					
7	Straw					
8	Vinyl					
9	Wood					
10	**Any other**					
	Wall covering (Interior) Materials					
11	Bricks					
12	Concrete sheets					
13	Composites					
14	Plaster board					
15	Tiles					
16	Wall paper					
17	Wood					
18	Partition Panels					
19	Acoustics					
20	Thermal Insulation					

12. What type of materials do you use for Water and Damp proofing?

S.No	Materials	Very Rarely	Rarely	Moderately	Often	Very Often
A	**Water proofing Materials**					
1	Asphalt					
2	Composites					
3	High Density Polyethylene (HDPE)					
4	Latex					
5	Membranes					
6	Metal					

7	Plastic					
8	Poly Vinyl Chloride (PVC)					
B	**Damp proofing**					
1	Acrylic polymers					
2	Asphalt					
3	Bricks					
4	Cement concrete					
5	Composites					
6	Glass fibre					
7	Metal sheets					
8	Mortar					
9	Polythene sheets					
11	Sheets and felts					
12	Slates					
13	Stone					
14	Stones					

13. What are the factors you would consider while selecting the materials?

S.No	Factors	Strongly Agree	Agree	Moderately Agree	Disagree	Strongly Disagree
1	Anti pest attack					
2	Appearance					
3	Cleanliness					
4	Clients Interest					
5	Cost factor					
6	Damp Proof					
7	Durable					
8	Ease of Maintenance					
9	Fire resistance					
10	Hardness					
11	Maintenance					
12	Recyclable material					
13	Repair flexibility					
14	Service life					
15	Smoothness					
16	Sound Insulation					
17	Thermal					

	Insulation					
18	Traffic levels					
19	Trends					
20	Light weight					
21	Green/Ecofriendly material					
22	**Any other**					

14. What are the other fibres you use in construction field?

Nylon	☐
Wool	☐
PET	☐
PE	☐
Acrylic	☐
Any other	☐

15. a Do you have awareness about Natural fibres used in construction field?

☐ Yes ☐ No

15.b If yes, what are the fibres you have come across?

Coir ☐
Sisal ☐
Jute ☐
Cotton ☐
Sisal ☐
Hemp ☐
Glass ☐
Kenaf ☐
Flax ☐
Others Specify. ☐

16a. In which all applications do you find natural fibres?

Flooring ☐

Wall covering ☐

Ceiling ☐

Others Specify ☐

17. What type of fabricated structure have you seen/used?

Nonwoven ☐

Composites ☐

Woven ☐

Fibres as such ☐

Any others ☐

18. Which type of structure do you find the most suitable for building interior construction/ decoration?

Nonwoven ☐

Composites ☐

Woven ☐

Fibres as such ☐

Any others ☐

19. Are the construction materials available in the local market?

☐ Yes ☐ No

20. a Do you prefer eco-friendly products for building construction

Preferable ☐

Somewhat preferable ☐

Mostly preferable ☐

Not preferable ☐

20.b If preferred, for what application you are using in the building construction?

Flooring ☐

Wall Covering ☐

Wall partition ☐

False roofing ☐

Any other ☐

20c If not preferred, give reasons

Cost ☐
Lack of Durability ☐

Labour ☐

Availability ☐

Awareness ☐

Any other ☐

21. Do you prefer the reusable materials for building application?

Preferable ☐

Somewhat preferable ☐

Mostly preferable ☐

Not preferable ☐

22. How much are you satisfied with the existing building materials and their supply ?

Aspects	**Very satisfied**	**Somewhat satisfied**	**Neither satisfied nor dissatisfied**	**Somewhat dissatisfied**	**Very dissatisfied**
Ease in Availability					
Cost effective					
Quality					
Durability					
Usability					
Service life					
Availability of natural product					
Labour intensive					
Any other					

23. What are the major challenges/ Problems you face in civil field regarding material?

Aspects	Strongly Agree	Agree	Moderately Agree	Disagree	Strongly Disagree
Reclaim of building materials					
dismissal disposal from building					
Lack of earth quake resilient structure					
Ageing of concrete					
Following Government policies					
Satisfying Client demands					
Indulging Green building concepts					
Lack of modern technology					
Checking for Safety measures					
Bringing Quality control					
Occurrence of Legal issues					
Reclaim of building materials					

24. What are the common criticisms received from customers about existing buildings?

Problems	Always	Sometimes	Not at all
Wall cavities			
Seepage			
Product life span			
Water leakage			
Pest and terminate problems			

Wall cracks			
Damage of door and window frames			
Floor damages			
Plumbing works			
Ageing of building			
Microbial attack			
Insect attack			
Voids due to poor workmanship			
Crumbling of plaster			
Re-plastering of walls as required			
Relaying cracked flooring at ground level			

25. Do you expect any special kind of material for construction?

□ Yes □ No

If yes, what is your expectation for construction materials?

Appendix II

Plant Authentication Certificates

भारतसरकार
GOVERNMENT OF INDIA
पर्यावरण, वन और जलवायु परिवर्तन मंत्रालय
MINISTRY OF ENVIRONMENT, FOREST & CLIMATE CHANGE
भारतीय वनस्पति सर्वेक्षण
BOTANICAL SURVEY OF INDIA

दक्षिणीक्षेत्रीयकेन्द्र / Southern Regional Centre
टी.एन.ए.यूकैम्पस/ T.N.A.U. Campus
लाउलीरोड/ Lawley Road
कोयंबत्तूर/ Coimbatore - 641 003

टेलीफोन / Phone: 0422-2432788, 2432123, 2432487
टेलीफक्स/ Telefax: 0422- 2432835
ई-मैल/E-mail id: sc@bsi.gov.in
bsisc@rediffmail.com

स. भा.व.स.द.क्षे.के./No.: BSI/SRC/5/23/2019/Tech /279

दिनांक/Date: 17th October 2019

पौधे प्रमाणीकरण प्रमाणपत्र /PLANT AUTHENTICATION CERTIFICATE

The plant specimen brought by you for authentication is identified as *Abutilon indicum* (L.) Sweet - MALVACEAE. The identified specimen is returned herewith for preservation in their College/ Department/ Institution Herbarium.

14/x/2019

डॉ.एम. यू. शरीफ़/Dr. M.U. Sharief
वैज्ञानिक 'ई' एवं कार्यालयाध्यक्ष/
Scientist 'E' &Head of Office

वैज्ञानिक 'ई' एवं कार्यालय अध्यक्ष
SCIENTIST 'E' & HEAD OF OFFIC
भारतीय वनस्पति सर्वेक्षण
BOTANICAL SURVEY OF INDIA
दक्षिणी क्षेत्रीय केन्द्र
SOUTHERN REGIONAL CENTRE
कोयम्बतूर / COIMBATORE - 641 00

14/10/19

सेवा में / To

Mrs. Thamarai Selvi
Ph.D Research scholar
Department of Textiles and Clothing
Avinashilingam Institute for Home Science
& Higher Education for Women
Coimbatore – 641 043

भारत सरकार
GOVERNMENT OF INDIA
पर्यावरण, वन और जलवायु परिवर्तन मंत्रालय
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दक्षिणी क्षेत्रीय केन्द्र / Southern Regional Centre
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ई-मैल /E-mail id: sc@bsi.gov.in
bsisc@rediffmail.com

सं. भा.व.स./द.क्षे.के./No.: BSI/SRC/5/23/2019/Tech. /281

दिनांक/Date: 14th November 2019

पौधे प्रमाणीकरण प्रमाणपत्र / PLANT AUTHENTICATION CERTIFICATE

The plant specimen brought by you for authentication is identified as *Agave americana* L. - ASPARAGACEAE. The identified sample is returned herewith for preservation in their College / Department / Institution Herbarium.

14/xi/2019

डॉ. एम. यू. शरीफ़/Dr. M.U. Sharief
वैज्ञानिक 'ई' एवं कार्यालयाध्यक्ष/
Scientist 'E' & Head of Office

वैज्ञानिक 'ई' एवं कार्यालय अध्यक्ष
SCIENTIST 'E' & HEAD OF OFFICE
भारतीय वनस्पति सर्वेक्षण
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SOUTHERN REGIONAL CENTRE
कोयम्बटूर / COIMBATORE - 641 003

14-11-19

सेवा में / To

Ms. S. Thamarai Selvi
Ph.D. Research Scholar
Department of Textiles and Clothing
Avinashilingam Institute for Home science
& Higher Education for Women
Coimbtaoe – 641 043

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bsisc@rediffmail.com

स. भा.व.स.द.क्षे.के./No.: BSI/SRC/5/23/2019/Tech /280

दिनांक/Date: 17[th] October 2019

पौधे प्रमाणीकरण प्रमाणपत्र /PLANT AUTHENTICATION CERTIFICATE

The plant specimen brought by you for authentication is identified as *Areca catechu* L. - ARECACEAE. The identified specimen is returned herewith for preservation in their College/ Department/ Institution Herbarium.

डॉ.एम. यू. शरीफ़/Dr. M.U. Sharief
वैज्ञानिक 'ई' एवं कार्यालयाध्यक्ष/
Scientist 'E' &Head of Office

वैज्ञानिक 'ई' एवं कार्यालय अध्यक्ष
SCIENTIST 'E' & HEAD OF OFFICE
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सेवा में / To

Mrs. Thamarai Selvi
Ph.D Research scholar
Department of Textiles and Clothing
Avinashilingam Institute for Home Science
& Higher Education for Women
Coimbatore – 641 043

Appendix III

Proforma for Obtaining Feedback of Untreated and Treated Fibre Samples on Visual Evaluation

S. No.	Sample Code	Colour			Lustre			Texture			General appearance		
		Bright	Medium	Dull	Good	Fair	Poor	Soft	Coarse	Very Coarse	Good	Fair	Poor
1	A												
2	B												
3	C												
4	D												
5	E												
6	F												
7	G												
8	H												
9	I												
10	J												
11	K												
12	L												
13	M												
14	N												
15	O												
16	P												
17	Q												
18	R												
19	S												
20	T												
21	U												

Appendix IV

Formula used to calculate Moisture Content, Regain and Water Absorption

Appendix IVa

Moisture Content

Formula used to calculate Moisture content

$$\text{Moisture content} - \frac{M_1 - M_2}{M_1} \times 100$$

M_1 – initial weight, M_2 – weight after drying

Appendix IVb

Moisture Regain

Formula used to calculate Moisture Regain (for fibre and nonwoven fabric samples)

The percentage of moisture in a material brought with standard atmosphere after partial drying and calculated as a percentage of the moisture free weight

Moisture Regain (%) = 100 × (Weight of moisture present/weight of oven dried sample) = 100 × (W1-W2)/ W2

where W1 is the actual weight of the sample; and W2, the oven dried weight of sample.

Appendix IV c

Water Absorption

Formula used to calculate Water Absorption (for fibre and nonwoven fabric samples)

Water absorption was determined using Equation.

$$\text{Water absorption (\%)} - \frac{M_1 - M_0}{M_0} \times 100$$

M1 is the mass of the sample after immersion and M_0 is the mass of the sample before immersion. This assessed for both of untreated and treated fibre samples.

Appendix V

Procedure for Soil burial test

The samples were frequently moistened using distilled water to maintain the humidity of the soil. The mean ambient temperature and relative humidity (RH) for the analysis were 26 ± 4 ◦C and 76 ± 4%, respectively, while the pH of the soil was 6.5. A mesh was used for wrapping the samples before being buried in the soil that would allow the moisture and microorganisms to access. Before the testing, all samples were dried at 105 ◦C and weighed which was recorded to determine the initial weight. All samples were soil-buried for specified durations of twenty, forty and sixty days. Subsequently, the respective samples were carefully taken out after the mentioned days of burial at specific intervals and impurities were mildly cleansed using distilled water. The degraded samples were oven dried and reweighed to acquire the final weight.

Appendix VI

Visual Evaluation of Soil buried Composite Samples

S.No	Samples	Colour				Texture				General appearance		
		Very Bright	Bright	Dull	Very Dull	Very Rough	Rough	Soft	Very Soft	Very Good	Good	Fair
1	A0											
	A20											
	A40											
	A60											
2	B0											
	B20											
	B40											
	B60											
3	C0											
	C20											
	C40											
	C60											
4	D0											
	D20											
	D40											
	D60											
5	E0											
	E20											
	E40											
	E60											

Appendix VII

Questionnaire To Elicit Information About Prepared Composite Slab

1. Name and Designation :

2. Address of the firm :

3. How do you feel about the developed fibre reinforced composite material?

S.No	Aspects	Excellent	Very Good	Good	Average	Not Satisfactory
a.	General appearance					
b.	Color					
c.	Texture					
d.	Strength					

4. Will this be suitable for building material? Yes /No
   If yes, rank the application order of the material

   a. False ceiling
   b. Wall panels and partitions
   c. Furniture
   d. Wooden shelves
   e. Decorative products
   f. Acoustic boards

5. Do you feel it is cost effective compared to commercially available products?

   Yes /No

6. Will this material have market potentiality? Yes/ No
   If yes, mention

7. Do you think the product is environment friendly?

   a. Yes b. No

   If yes,

8. Give the main reason for preferring eco friendly materials?

a. Enhance a quality of life

b. Environmental protection responsibility

c. potential increase of product valued

d. getting high level of satisfaction

9. Would you recommend our product to other builders?

a. Yes b. No

10. Any other suggestions:

Appendix VIII

Tensile Strength and Elongation of *Abutilon indicum* Fibres

Zwick / Roell

30.01.18

Test report

Job no. : C1700607-2
Tester : JA
Specimen type : FIBRE SAMPLE
Notes : SAMPLE ID-AB I

Pre-load : 0.1 N
Speed, Young's modulus : 1 mm/min
Test speed : 5 mm/min

Test results:

No.	F_{max} cN	W to break Nmm	Elongation %
1	678.93	1.91	1.27
2	303.89	0.52	0.66
3	440.76	1.21	1.07
4	435.14	1.50	1.27
5	422.21	1.39	1.39
6	331.08	0.75	1.06
7	411.15	1.11	0.84
8	261.74	0.67	1.13
9	767.17	2.36	1.54
10	453.80	1.48	1.46
11	811.78	2.27	1.31
12	1142.16	5.18	1.52
13	520.85	1.92	1.31
14	938.52	2.49	1.17
15	931.76	2.77	1.13

No.	F_{max} cN	W to break Nmm	Elongation %
16	651.55	1.78	1.13
17	882.50	3.01	1.46
18	623.54	1.49	1.08
19	796.73	2.32	1.26
20	714.74	2.46	1.51
21	444.44	1.66	1.41
22	531.35	1.74	1.39
23	418.85	1.54	1.64
24	489.10	1.58	1.29
25	841.26	3.11	1.48
26	671.64	1.87	1.17
27	805.17	2.43	1.22
28	383.95	1.27	1.24
29	388.37	1.13	1.24
30	537.19	1.44	1.16

Statistics:

Series n = 30	F_{max} cN	W to break Nmm	Elongation %
$\bar{x}$	601.04	1.88	1.26
s	222.00	0.91	0.21
ν [%]	36.94	48.20	16.62

Appendix VIIIa

Consolidated results for chemical constituents of fibres

Chemical Constituents (%)					
S.No	**Fibre Samples**	**Cellulose**	**Lignin**	**Wax**	**Ash**
1	AUN	67.95	24.17	0.18	2.2
2	AC1	64.3	22.81	0.11	3.11
3	AC2	**62.9**	25.17	0.29	0.52
4	AC3	65.03	22.55	0.17	1.48
5	AE1	61.25	26.16	0.44	1.06
6	AE2	70.72	14.65	0.33	1.24
7	AE3	60.43	25.24	0.22	1.27
8	BUN	70.77	13.59	0.29	3.1
9	BC1	87.95	13.46	**0.21**	1.99
10	BC2	69.37	**10.86**	0.36	**1.13**
11	BC3	**68.3**	17.85	0.23	1.72
12	BE1	77.76	9.96	**0.27**	**1.05**
13	BE2	**73.85**	10.38	0.48	2.06
14	BE3	78.4	**9.44**	0.45	1.29
15	CUN	57.79	27.92	0.44	2.22
16	CC1	**52.9**	31.78	0.23	2.34
17	CC2	62.73	**23.03**	0.30	**1.07**
18	CC3	63.17	24.48	**0.18**	1.95
19	CE1	67.85	20.73	**0.28**	1.57
20	CE2	**56.88**	**18.70**	0.35	**0.99**
21	CE3	58.39	26.94	0.39	1.87

Appendix VIII b

Results obtained for moisture content from The South India Textile Research Association

THE SOUTH INDIA TEXTILE RESEARCH ASSOCIATION
SITRA CHEMICAL LABORATORY
13/37 Avanashi Road, Aerodrome Post, Coimbatore - 641 014, INDIA
Ph : (0422) 2574367-9, 4215334, 4215328 Fax : (0422) 2571896, 4215300
Email: chem@sitra.org.in, info@sitra.org.in Website:http:// www.sitra.org.in
Address all correspondence to the Director

ThamaraiselviS Ref : Dt.16.07.18

Fibre Chemical Composition Testing	G1801062-1 SAK	G1801062-2 AAK	G1801062-3 SACE	G1801062-4 AACE
Cellulose Content, % (Method: SITRA/TC/FCC/01)	71.95	52.9	69.37	62.73
Lignin Content, % (Method: SITRA/TC/FCC/02)	13.46	32.1	10.86	23.03
Wax Content, % (Method: SITRA/TC/GT/09)	0.21	0.23	0.36	0.30
Ash Content (on dry basis), % (Method: IS 199)	1.99	2.51	1.13	1.07
Moisture Content, % (Method: IS 199)	12.17	12.08	18.04	12.70
Density, g/cc (Method: SITRA/TC/FCC/03)	1.381	1.242	1.236	1.384
Fibre Chemical Composition Testing	**G1801062-5 ABACE**	**G1801062-6 SB**	**G1801062-7 AB**	**G1801062-8 ABB**
Cellulose Content, % (Method: SITRA/TC/FCC/01)	62.9	68.3	63.17	65.03
Lignin Content, % (Method: SITRA/TC/FCC/02)	25.17	17.85	24.48	22.55
Wax Content, % (Method: SITRA/TC/GT/09)	0.29	0.33	0.18	0.17
Ash Content (on dry basis), % (Method: IS 199)	0.52	1.72	1.95	1.48
Moisture Content, % (Method: IS 199)	10.99	11.61	10.05	10.56
Density, g/cc (Method: SITRA/TC/FCC/03)	1.372	1.414	1.82	1.324
Fibre Chemical Composition Testing	**G1801062-9 SC**	**G1801062-10 AC**	**G1801062-11 ABC**	**G1801062-12 SP**
Cellulose Content, % (Method: SITRA/TC/FCC/01)	77.76	67.85	61.25	73.85
Lignin Content, % (Method:	9.96	20.73	26.16	10.38

Appendix VIIIc

Results obtained for moisture content from The South India Textile Research Association

THE SOUTH INDIA TEXTILE RESEARCH ASSOCIATION
SITRA CHEMICAL LABORATORY
13/37 Avanashi Road, Aerodrome Post, Coimbatore - 641 014, INDIA
Ph : (0422) 2574367-9, 4215334, 4215328 Fax : (0422) 2571896, 4215300
Email: chem@sitra.org.in, info@sitra.org.in Website:http:// www.sitra.org.in
Address all correspondence to the Director

ThamaraiselviS Ref : Dt.16.07.18

SITRA/TC/FCC/02)				
Wax Content, % (Method: SITRA/TC/GT/09)	0.27	0.28	0.44	0.48
Ash Content (on dry basis), % (Method: IS 199)	1.05	1.57	1.06	2.06
Moisture Content, % (Method: IS 199)	10.75	9.38	10.89	13.02
Density, g/cc (Method: SITRA/TC/FCC/03)	1.413	1.375	1.400	1.378
Fibre Chemical Composition Testing	**G1801062-13 AP**	**G1801062-14 ABP**	**G1801062-15 SCP**	**G1801062-16 ACP**
Cellulose Content, % (Method: SITRA/TC/FCC/01)	56.88	70.72	78.4	58.39
Lignin Content, % (Method: SITRA/TC/FCC/02)	18.70	14.65	9.44	26.94
Wax Content, % (Method: SITRA/TC/GT/09)	0.35	0.33	0.45	0.39
Ash Content (on dry basis), % (Method: IS 199)	0.99	1.24	1.29	1.87
Moisture Content, % (Method: IS 199)	22.88	12.85	10.22	12.21
Density, g/cc (Method: SITRA/TC/FCC/03)	1.412	1.350	1.363	1.420
Fibre Chemical Composition Testing	**G1801062-17 ABCP**	**G1801062-18 ABAK**	**G1801062-19 UNS**	**G1801062-20 UNA**
Cellulose Content, % (Method: SITRA/TC/FCC/01)	60.43	60.6	70.77	57.79
Lignin Content, % (Method: SITRA/TC/FCC/02)	25.24	24.81	13.59	27.92
Wax Content, % (Method: SITRA/TC/GT/09)	0.22	0.11	0.29	0.44
Ash Content (on dry basis), % (Method: IS 199)	1.27	3.11	3.1	2.22

Appendix VIIId

Results obtained for moisture content from The South India Textile Research Association

THE SOUTH INDIA TEXTILE RESEARCH ASSOCIATION
SITRA CHEMICAL LABORATORY
13/37 Avanashi Road, Aerodrome Post, Coimbatore - 641 014, INDIA
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Email: chem@sitra.org.in, info@sitra.org.in Website:http:// www.sitra.org.in
Address all correspondence to the Director

ThamaraiselviS *Ref : Dt.10.07.18*

Moisture Content, % (Method: IS 199)	12.6	11.6	12.02	11.43
Density, g/cc (Method: SITRA/TC/FCC/03)	1.436	1.45	1.37	1.46
Fibre Chemical Composition Testing	**G1801062-21 UNAB**			
Cellulose Content, % (Method: SITRA/TC/FCC/01)	61.2			
Lignin Content, % (Method: SITRA/TC/FCC/02)	24.17			
Wax Content, % (Method: SITRA/TC/GT/09)	0.18			
Ash Content (on dry basis), % (Method: IS 199)	2.2			
Moisture Content, % (Method: IS 199)	12.10			
Density, g/cc (Method: SITRA/TC/FCC/03)	1.45			

- End of Report -

Ref: Dt 10.07.2018

Appendix VIII e

The altered nomenclature used for the fibre samples

S.No	Laboratory nomenclature	Long form for sample names	Altered nomenclature
1	UNAB	Untreated *Abutilon indicum*	AUN
2	ABAK	*Abutilon indicum* after alkalization	AC1
3	ABACE	*Abutilon indicum* after acetylation	AC2
4	ABB	*Abutilon indicum* after benzoylation	AC3
5	ABC	*Abutilon indicum* after Cellulase treatment	AE1
6	ABP	*Abutilon indicum* after Pectinase treatment	AE2
7	ABCP	*Abutilon indicum* after Cellulase and Pectinase treatment	AE3
8	UNS	Untreated *Agave Americana*	BUN
9	SAK	*Agave Americana* after alkalization	BC1
10	SACE	*Agave Americana* after acetylation	BC2
11	SB	*Agave Americana* after benzoylation	BC3
12	SC	*Agave Americana* after Cellulase treatment	BE1
13	SP	*Agave Americana* after Pectinase treatment	BE2
14	SCP	*Agave Americana* after Cellulase and Pectinase treatment	BE3
15	UNA	Untreated *Areca catechu*	CUN
16	AAK	*Areca catechu* after alkalization	CC1
17	AACE	*Areca catechu* after acetylation	CC2
18	AB	*Areca catechu* after benzoylation	CC3
19	AC	*Areca catechu* after Cellulase treatment	CE1
20	AP	*Areca catechu* after Pectinase treatment	CE2
21	ACP	*Areca catechu* after Cellulase and Pectinase treatment	CE3

Appendix IX

FTIR of *Abutilon indicum* Fibre Samples

S.no	Sample	Absorption (cm^{-1})	Assignment	Functional Group
1	AUN	3741.90	O-H stretching	Hydroxyl group
		3626.17	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1743.65	C=O stretching	Ester
		1365.60	O-H bending	Hydroxyl group
2	AC1	3865.35	O-H stretching	Hydroxyl group
		3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1743.65	C=O stretching	Ester
		1365.60	O-H bending	Hydroxyl group
3	AC2	3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1735.93	C=O stretching	Ester
		1604.77	C=C stretching	Alkene
		1365.60	O-H bending	Hydroxyl group
4	AC3	3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		2129.41	O=C=O stretching	Carbon dioxide
		1365.60	O-H bending	Hydroxyl group
5	AE1	3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1743.65	C=O stretching	Ester
		1365.60	O-H bending	Hydroxyl group
6	AE2	3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1743.65	C=O stretching	Ester
		1365.60	O-H bending	Hydroxyl group
		1226.73	C-F stretching	Fluoro compound
7	AE3	3741.90	O-H stretching	Hydroxyl group
		2360.87	O=C=O stretching	Carbon dioxide
		1735.93	C=O stretching	Ester
		1365.60	O-H bending	Hydroxyl group
		1226.73	C-F stretching	Fluoro compound

Appendix X

Air Permeability of Nonwoven Fabric Samples

BHARATHIAR UNIVERSITY
COIMBATORE – 641 046, TAMILNADU, INDIA

State University | Accredited with "A" Grade by NAAC | Ranked 13th among Indian Universities by MHRD-NIRF

DEPARTMENT OF TEXTILES AND APPAREL DESIGN

TEXTILE TESTING LABORATORY

TEST REPORT

Report No: TAD/R/012
Sample code: CHABS

Date: 11.03.2021
Sample details: Non woven

Name: Ms.S.Thamarai Selvi, Avinashilingam Institute

Air Permeability Test

S. No.	Manometer Reading	Rotameter Reading		
		R1	R2	R3
1.	5.0	2200	600	60
2.	5.5	2600	600	60
3.	5.9	2600	600	60
4.	6.0	2600	600	60
5.	5.2	2200	600	60
6.	5.8	2600	600	60
7.	5.4	2400	600	60
8.	6.0	2600	600	60
9.	5.3	2400	600	60
10.	5.7	2600	600	60
Total		24800	6000	600
Average		**2480**	**600**	**60**

Air Permeability $R = r \times 1000 / 60 \times 60 \times A$

Where, r- Rotameter reading in liter per hour, A-Treat Area (4 or 10 cm^2)

$r = 2480 + 600 + 60 = 3140$LPH, $A = 10cm^2$

Calculation

$$R = \frac{3140 \times 1000}{3600 \times 10}$$

Air Permeability R= 87.22 $cm^3/sec/cm^2$

Signature of Lab incharge

Dr. K. AMUTHA
Assistant Professor
Dept. of Textiles and Apparel Design
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Signature of HOD

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Appendix XI

FTIR of Composites

S.no	Sample	Wave number (cm^{-1})	Bond and Functional Group
1	EP	1728.22	C=C, Stretch Alkanes (Lignin)
		1427.32	C-C, Stretch Aromatic amines
		1357.89	C-H, Bending Nitro compound, Methane
		1234.44	C-O, Stretch Aromatic amines
		1026.13	C-N, Stretch Aliphatic amines
2	CABSE	1743.65	C=O stretching of carboxylic acid or ester
		1705.07	C=O Carboxylic acid
		1597.06	C-C Aromatic compound, Alkene
		1512.19	C-C Aromatic compound, Alkene
		1126.43	C-X Fluoride
3	CASE	1743.65	C=O Ester
		1712.79	Carboxylic acid
		1597.06	C-C Aromatic compound, Alkene
		1388.75	C-O Alcohols, ethers, esters, Carboxylic acids, anhydrides
		1126.43	C-X Fluoride
4	CAABE	1743.65	C=O Ester
		1705.07	C=O Carboxylic acid
		1597.06	C-C Aromatic compound, Alkene
		1388.75	C-O Alcohols, ethers, esters, Carboxylic acids, anhydrides
		1123.43	C-X Fluoride
5	EAABSE	1743.65	C=O Ester
		1705.07	C=O Carboxylic acid
		1512.19	C-C Aromatic compound, Alkene
		1126.43	C-X Fluoride

Appendix XII

Standard deviation values of mechanical properties of composite structures

Composite Mechanical Properties

S.No	Samples	Standard deviation values of mechanical properties of composite structures					
		Tensile Strength	Elongation	Flexural Strength	Impact Strength	Compressive Strength	Shore D Hardness
1	EP	5.0349	1.36328	6.60654	0.05701	2.26851	0.16
2	EAABSE	0.59665	1.14146	16.79956	0.13038	1.84658	2.41
3	CASE	5.95442	0.57352	7.38709	0.09354	4.04394	1.58
4	CAABE	7.08662	0.38626	2.432	0.07416	3.86617	1.58
5	CABSE	6.48396	0.43114	4.57093	0.1	2.70518	0.16

Appendix XIII

Cost Comparison of Prepared Composite Slab with Commercially Available Panel

S.No	Cost Particulars	Product Rate (Rs) Unit price
1	Fibre	45
2	Fibre Treatment	50
3	Nonwoven Fabrication	45
4	Resin	110
5	Cutting and Shaping	20
6	Labour involved and others	100
	Total cost of prepared composite slab – (rate/sq. ft)	**370**
	Commercially available acoustic panel – (rate/sq. ft)	**345**
	Commercially available acoustic fabric panel - – (rate/sq. ft)	**220**
	Commercially available acoustic hexagonal panel - – (rate/sq. ft)	**795**

5

Avinashilingam Institute for HomeScience and Higher Education for Women

(Deemed to be University Estd. u/s 3 of UGC Act 1956, Category 'A' by MHRD
Re-accredited with A++ Grade by NAAC. CGPA 3.65/4, Category I by UGC
Coimbatore - 641 043, Tamil Nadu, India

Appendix L2

(Item No 5 of Check List) Details of Research Publications

S.No	Article	Journal	Other Details Vol/No/Page No/ Year	Published in UGC-CARE / Scopus Indexed/ Web of Science
1	Impact of Chemical Treatment on Surface Modification of Agave americana fibres for composite Application - A futuristic Approach.	Journal of Natural fibres	20, Issue 1 2023.	Scopus Indexed.
2	Effect of chemical and biological treatments on Agave americana Yarn braid reinforced composite structures	Materials Chemistry and physics	doi.org/10.1016 2023	Scopus Indexed.

*Proof of list of Journals from Internet to be attached along with copies of reprints.

Scholar :

Supervisor :

20/1/23

Checked By:

HoD/Dean of Respective School

Journal of Natural Fibers

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/wjnf20

Impact of Chemical Treatment on Surface Modification of Agave Americana Fibres for Composite Application – A Futuristic Approach

S. Thamarai Selvi, R. Sunitha, L. Ammayappan & C. Prakash

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To link to this article: https://doi.org/10.1080/15440478.2022.2142726

JOURNAL OF NATURAL FIBERS
2023, VOL. 20, NO. 1, 2142726
https://doi.org/10.1080/15440478.2022.2142726

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Impact of Chemical Treatment on Surface Modification of Agave Americana Fibres for Composite Application – A Futuristic Approach

S. Thamarai Selvi[a], R. Sunitha[a], L. Ammayappan[b], and C. Prakash[c]

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ABSTRACT

As a futuristic approach an attempt has been made in this study to improve the compatibility between reinforcement and polymer matrix in composites by various chemical treatments. The adhesion of these can be achieved by modifying the surface using the chemical treatments. The general appearance of the *Agave Americana* fibers which underwent benzoylation was rated to be good in visual evaluation. The chemical treatments increased lignin content with 31.3% on benzoylation but decreased by both acetylation and alkylation treatments with 20.08% and 0.95% respectively. Moisture content was decreased in the fiber samples by benzylation and water absorption property decreased on acetylation and alkylation which would assist in fiber matrix compatibility. The breaking strength increased on benzoylation, and elongation increased on alkylation. Among the three chemical treatments the maximum degradation of fibers occurred at the highest temperature of 390°C with mass loss of 98.2% by benzoylation. Thermal stability improved in all the chemical treated fiber samples. Morphological analysis exhibited the removal of gummy substances and had improved roughness on the surface of fibers. The novelty is brought in the study by the fiber selection and analysis made using varied chemicals.

摘要

作为未来的方法，本研究试图通过各种化学处理改善复合材料中增强材料和聚合物基体之间的相容性. 可以通过使用化学处理对表面进行改性来实现这些材料的粘附. 经过苯甲酰化处理的龙舌兰纤维的总体外观在视觉评价中被评为良好. 化学处理使苯甲酰化木质素含量增加31.3%，而乙酰化和烷基化处理木质素含量分别降低20.08%和0.95%. 苄基化降低了纤维样品中的水分含量，乙酰化和烷基化降低了吸水性能，这有助于纤维基质的相容性. 苯甲酰化时断裂强度增加，烷基化时伸长率增加. 在三种化学处理中，纤维的最大降解发生在最高温度390°C，苯甲酰化导致的质量损失为98.2%. 所有化学处理过的纤维样品的热稳定性都有所提高. 形态分析表明，去除了胶质物质，并改善了纤维表面的粗糙度. 通过使用各种化学物质进行纤维选择和分析，这项研究带来了新颖性.

KEYWORDS

Agave Americana; chemical treatment; lignocellulosic fibers; natural fibers; surface modification; surface morphology

关键词

木质纤维素纤维; 美国龙舌兰; 化学处理; 天然纤维; 表面改性; 表面形貌

Introduction

The fibers obtained from natural source offer many technical and ecological benefits for its utilization as reinforcements in composites (Ramesh et al. 2021a, 2021b). The natural fibers are classified as bast fibers namely flax, jute, hemp, kenaf and ramie; leaf fibers such as agave, abaca, and pineapple; fruit

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and seed fibers such as coir, cotton and kapok, core fibers such as kenaf, hemp and jute, grass and reed fibers namely bamboo, elephant grass, wheat, corn and rice and other fibers from wood and roots (Asim et al. 2015). Natural fibers such as sisal, coir, jute, palm, pineapple have gained potentiality as reinforcements in composites for automotive, aerospace, sports, marine, electrical, construction and household appliances (Ramesh et al. 2014; Ramesh, Rajeshkumar, and Bhuvaneswari 2022).

Sisal fiber which belongs to Agavaceae family is a stable, strong and versatile one which finds important place in polymer composites manufacturing. The natural fiber composites are advantageous as they are renewable resources and need low energy during their manufacturing process. These are not only biodegradable and renewable but also are advantageous because they are their lightweight, economic, high strength and safe (Asma et al. 2020). High moisture absorption is the main disadvantage of using natural fibers in composite manufacturing (Gupta and Rao 2016).These characteristics are primarily due to their inherent heterogeneous content such as cellulose, hemicellulose, starch, lignin, pectin, waxes, fats and other polar compounds (Mortazavi and Kamali Moghaddam 2010). Poor interface adhesion between natural fibers and polymer matrices is due to the differences in the wettability of natural fibers and the polymer matrix. This also reduces the efficiency of stress distribution from the matrix to the fiber and simultaneously diminishes the mechanical properties of such natural fiber-reinforced composites (Punyamurthy et al. 2012). So, the alkali treatment with sodium hydroxide solution is preferred for modifying the properties of natural fibers to improve interfacial bonding between the fibers and the matrices in the composite manufacturing. Alkalization is used to improve interfacial strength between fiber and matrix and dynamic flexural modulus of the composites (Saha et al. 2010).

Materials and methods

Collection of Agave americana *leaves*

Agave americana commonly known as *American aloe or maguey belongs to monocotyledonous family called Agavaceae* (Hulle, Kadole, and Katkar 2015). *Agave americana* plants are locally available in and around the districts of Tamil Nadu. These plants are xerophytic, monocarp, semi-perennial and leaf fiber producing plant. The plant comprises short stem which bears rosette of leaves that attains a length of 1–1.5 m or more. The leaves are thick, fleshy and often covered with waxy layer, typical characteristics of Xerophytic plants. A healthy sisal plant produces about 200–250 leaves during its 10–12 years life span, after which it produces a long flowering axis. These fibers could be utilized for technical applications (Kolte, Daberao, and Miss Sharma 2012). The leaves at different stages of maturity were collected and the fibers were extracted. The extracted fibers were analyzed for strength and elongation. The stage, that yielded the strongest fibers, was selected for further study. Though it was known that the plant was *Agave americana*, through literature reviews, it was confirmed to be the same in the Botanical Survey of India, Tamil Nadu Agricultural University, Coimbatore, by submitting the specimen of the leaf and flower of the plant (Figure 1)

fiber extraction

The mechanical process of decortication was used for fiber extraction from the leaves of Agave Americana as the leaves ware fleshy and need the mechanical decortication for separation of the fibers (El Oudiani et al. 2008). The leaves of equal length were arranged together and fed into the decorticator. As the drum rotated, three to five leaves were fed between the drum and the backing plate. By the crushing, beating and pulling actions, the pulpy material was removed and when it was half way through, the leaves were pulled out slowly. Then the other half was fed in the same manner as before. The fibers obtained were washed, dried and combed when slightly wet, using a suitable brush. Combing was done to separate the short fibers from the long fibers.

Figure 1. *Agave americana* plants.

Fiber treatments

The *Agave Americana* fibers were modified to make it suitable for manufacturing composites as a futuristic approach. The chemical treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites. The surface roughness is also increased by the alkalization which is caused by the disruption of hydrogen bonding in the network structure (Mohanty, Misra, and Drzal 2001).The major problem of biofiber polymer composite is the interfacial bonding of fiber and polymer caused by the hydrophilic nature of natural fiber against the hydrophobic nature of polymer which can be improved by chemical surface modification (Figure 2) (Mahjoub, Bin Mohamad Yatim, and Mohd Sam 2013).

Alkalization

The *Agave Americana* fibers were alkalized by immersing them in a 5% aqueous NaOH solution for 24 hours at room temperature as this chemical treatment would improve the fiber-matrix interaction and thermal behavior of composites in future (Amjad et al. 2021; Gupta, Gond, and Bharti 2018). The fibers were then extensively cleaned with distilled water to eliminate any traces of chemicals and neutralized (Li, Tabil, and Panigrahi 2007; Mishra et al. 2001).

Acetylation process

The *Agave Americana* fibers were soaked in demineralized water for an hour, then filtered and placed in a round bottom flask with acetylating solution comprising of 250 mL toluene, 125 mL acetic anhydride, and a little amount of catalyst perchloric acid. This process lasted for one hour and was carried out at a temperature of 60°C. The fibers were then rinsed with distilled water until it was acid-free and air dried, as described (Chubuike et al. 2017).

Benzoylation treatment

Benzoylation is a chemical treatment used to alter the surface of fibers (Atiqah et al. 2018). To activate the hydroxyl groups of the cellulose and lignin the *Agave americana* fibers were initially pretreated with alkaline. Then the fiber was suspended in 10% NaOH and benzoyl chloride solution for 15 min.

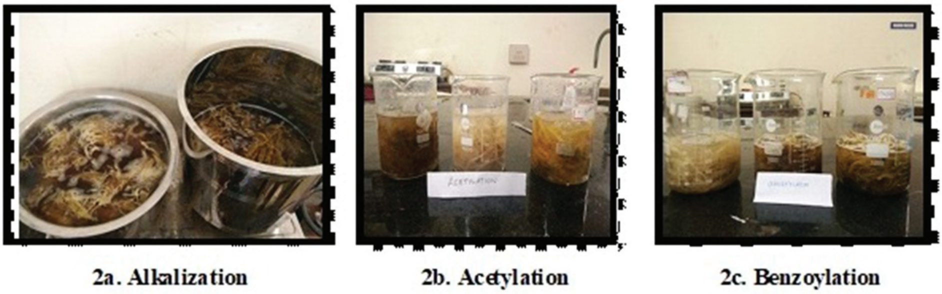

Figure 2. Fiber treatments.

Benzoyl chloride was used for reducing the hydrophobicity of the fiber and to enhance the fiber-matrix adhesion which ultimately would improve the tensile properties of composite structures. Then the fibers were soaked in ethanol for one hour to remove the traces of benzoyl chloride and was washed with water and dried for 24 hours in the oven at 80°C.

Evaluation of fibers

The extracted *Agave Americana* fibers were analyzed for force, elongation, time of rupture and length. The treated and untreated fibers were subjectively and objectively assessed for various essential properties.

Length and diameter

The length of the fiber is the distance between the fiber ends when a tension just sufficient to remove the crimp has been applied. So each fiber sample was straightened over a suitable scale and the length was measured directly. This procedure was followed for fifty fibers of *Agave americana* and the average was taken and recorded as the length of the fiber. The diameter of *Agave americana* fibers was analyzed using projection microscope. About 10 samples were viewed and noted.

Force, elongation and time of rupture

The *Agave Americana* fibers at various stages of maturity were tested for the properties of force, elongation and time of rupture in SITRA. Techno Statimat 4 Test Standard Tensile Tester which works on the principle of constant rate of extension was used. The fiber samples were taken randomly and subjected to testing. The gauge length was adjusted to 200 mm and test speed was kept at 208 mm/min. Since it was computerized, suitable keys were pressed for the movements of the jaws. Twenty samples were tested and the readings were automatically recorded in the system and output was obtained. The same procedure was repeated for the fibers at various stages of maturity.

Visual evaluation

The untreated and treated *Agave Americana* fiber samples (Figure 3) were subjected to visual evaluations which were judged by panel of judges numbering fifty. The samples were evaluated for the aspects namely color, luster, texture and general appearance individually by displaying them.

Spectrophotometric analysis

Measurement related to visual observations poses problem as different observers judge a sample differently in assessing the deviation in lightness, hue and saturation by personal preferences (Gupta, Kulkarni, and Gulrajani 1986). To prevent the human perceptions, the objective method of measuring color was done using spectrophotometer. The light reflected from the material is collected in an integration sphere, normalized to the source light of the reflectance and calibrated with the measurement of a pure white standard and black box over the entire wavelength spectrum of visible light. The mass of fibers were placed into a compression cell such that it does not protrude into the sphere and a constant amount of pressure was applied to avoid errors due to the gaps formed between fibers.

fiber strength, chemical constituents and moisture related properties

The fire chemical constituents and moisture behavior are very much essential for the fiber to be utilized for manufacturing composites. These fiber properties were assessed as per standards in SITRA laboratory. The chemical constituents namely cellulose lignin, wax and ash present in the fibers were observed by in-house method in SITRA laboratory. Also, the tensile strength and elongation were carried out by using Zwick Roell tensile testing machine in SITRA laboratory as per standards at 5 mm gauge length and 50 in speed. Thus, the tensile properties of the fibers were also analyzed to obtain information about the state of the fiber bundles specifically to understand the influence of treatment on fiber properties.

Water absorption

Many considerations have to be taken into account in the design of natural fiber composites. One of the most important issues is the degrading behavior of the composites exposed to environmental conditions such as humidity, sunlight, and microorganisms. Also, the poor resistance of the fibers to water absorption can have undesirable effects on the mechanical properties and the dimensional stability of the composites. Therefore, it is important to study in detail the water absorption behavior in order to estimate not only the consequences that the water absorbed may have, but also the durability of natural fibers composites aged under water.

Natural fibers are prone to water absorption due to their chemical constituents and hydrophilic properties. The water absorption behavior of *Agave americana* fibers, both untreated and treated, was examined. Individual fiber samples were bundled and dried in a hot air oven at 60°C for 24 hours before being weighed in an electronic balance with an accuracy of four decimals. This was immersed in water and kept in a humidity chamber at room temperature for 24 hours. Then the fiber bundles were

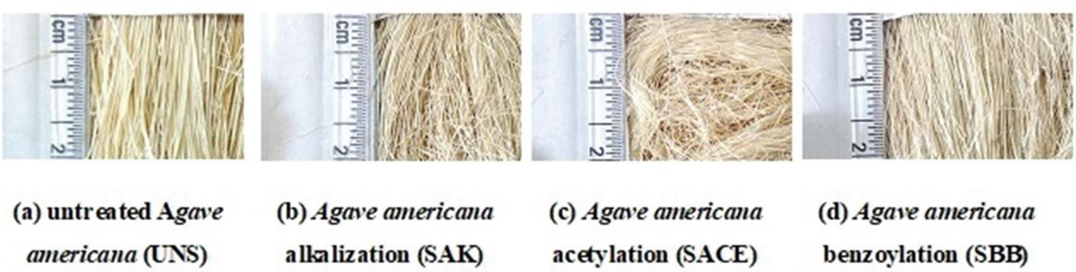

Figure 3. Images of fibers.

Table 1. Nomenclature of samples.

S.No.	Samples	Nomenclature
1	UNS	*Agave Americana* fiber samples – Untreated
2	SAK	*Agave Americana* fiber samples – Alkalization
3	SACE	*Agave Americana* fiber samples – Acetylation
4	SB	*Agave Americana* fiber samples – Benzoylation

removed from the humid environment, wiped with filter paper and weighed for calculating the percentage of water absorption.

Water absorption (%) = W_1-W_2/W_2x100

where W_1 is the mass after immersion to water, and the W_2 is the mass of sample before immersion.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) is very much essential test to understand the thermal stability of natural fibers throughout the heating process based on mass change; as a result, it is critical to understand the significant thermal stability of natural fibers at high temperatures of 220°C for at least a few minutes. Studies have reported that natural fibers are sensitive to temperature and complete thermal degradation is expected to occurr at temperatures of 400 ∘C and above (MohdRadzuan et al. 2021).

The thermal behavior is also of practical interest for conditions associated with temperatures above the ambient, as in fire damage, curing or process involving heating procedures. In fact, several works also assessed distinct thermal responses, particularly in terms of thermogravimetric properties of natural fiber polymer composites (Monteiro et al. 2012). The analyzer used for determining the thermal stability of untreated and treated *Agave americama* fibers was EXSTAR/6300. fiber samples weighing one milligram were placed in an alumina pan and heated between 50 and 450 degrees Celsius in a nitrogen gas environment. Thermo gravimetric analysis was used to investigate the fiber's thermal resilience and weight loss when exposed to higher temperatures. The TGA curves of untreated and treated *Agave americana* fiber samples obtained were recorded.

Morphological analysis

For analyzing the morphology of treated and untreated *Agave americana* fibers the scanning electron microscopic study was used. This was carried out using TESCAN-MIRA3 XMU scanning electron microscope for obtaining images of the fiber surface at various magnifications to evaluate the effect of chemical treatments on the fiber morphology and the best image was selected for comparison.

Nomenclature of samples

The untreated and treated fiber samples are designated as expressed in the Table 1.

Results and discussion

The results obtained in the tests carried out for the *Agave Americana* fibers are expressed under the following heads.

Length and diameter

Fiber length is an important physical parameter. The mean length of the *Agave americana* fibers obtained from the leaves of matured plants was found to be 100,125 centimeters with an average of 110 centimeters. The diameter of the fibers ranged from 150 μm to 300 μm. The average was found to be 235 μm.

Fiber characteristic based on various stages of maturity

Agave Americana fiber characteristics based on various stages of maturity namely matured, moderate and tender are given in Table 2.

Table 2 reveals that the force required to rupture the fibers was found to be the highest in matured fibers of 13.15n. The elongation percentage of matured fibers ranged between 4.04% and 7.25% with an average of 5.89% which is the highest among the three fiber samples. The time taken by the matured fibers to rupture was the highest of about 3.53 seconds. Hence matured fibers were proved to be the strongest of all the three stages assessed. Hence the matured fibers were ustilised for further study.

Visual evaluation

The results obtained by visual evaluation of the treated and untreated *Agave americana* fibers are presented in the Table 3.

From Table 3, it is clear that the *Agave americana* sample UNS exhibited medium color by 83% of judges. The same was noted in the chemical treated sample SAK also with maximum rating among the three treated samples followed by the samples SACE and SB with 77% and 70% of judges. The luster was reported to be fair by the maximum of 83% of judges in sample UNS. And poor luster was noted in sample SAK as per the judgment of 50% of judges, the sample SACE was assessed to be fair by 60% of judges and as good by 43% of judges. The texture was observed to be coarse in sample UNS as reported by 67% of judges. Among the chemical treated samples the maximum of 73% of judges expressed that the sample SAK was soft followed by the samples SACE and SB with 67 and 53% of judges. As for the general appearance the sample UNS was noted to have fair general appearance by the maximum of 83% of judges. This was noted in samples SAK and SACE with 67% and 57% of judges. The general appearance was noted to be good as assessed by 60% of judges in sample SB among the chemical treated samples. Hence it could be concluded that the general appearance was good in samples **SB** on chemical treatment.

Spectrophotometric analysis of treated and untreated Agave Americana *fibers*

The color indices results obtained for the *Agave Americana* fibers are presented in the Table 4.

From Table 4, it is clear that the chemical treated samples increased in the yellowness index over the untreated sample UNS (41.279) of which it was highest in the sample SACE (46.482) followed by the samples SAK (46.097) and SB (43.401). The whiteness index was noted to be minimum in sample SAK on chemical treatment. The brightness index was

Table 2. Fiber characteristic based on various stages of maturity.

S.No.	Leaf Stages	Force (n*)			Elongation (per cent)			Time of rupture (Seconds)
		Minimum	Maximum	Average	Minimum	Maximum	Average	
1	Matured	8.40	19.08	13.15	4.04	7.25	5.89	3.53
2	Moderate	3.73	9.84	6.95	1.60	5.88	3.92	2.42
3	Tender	2.03	11.19	6.45	1.20	5.63	3.81	2.34

Note: *- Results obtained in grams were converted into newton.

Table 3. Visual Evaluation of Treated and Untreated *Agave americana* fibers.

S. No.	Sample Code	Colour (%)			Luster (%)			Texture (%)			General appearance (%)		
		Bright	Medium	Dull	Good	Fair	Poor	Soft	Coarse	V. Coarse	Good	Fair	Poor
1	UNS	17	83	0	0	83	17	0	67	33	0	83	17
2	SAK	0	83	17	17	33	50	73	17	10	17	67	17
3	SACE	23	77	0	30	60	10	67	23	10	40	57	3
4	SB	20	70	10	43	17	40	53	27	20	60	20	20

observed to be the maximum in sample SACE (30.306) followed by samples SB (28.438) and SAK (25.428). The chemical treated samples in the objective assessment about brightness index was the highest in sample SACE followed by the sample SB which is also observed in the subjective analysis also.

Chemical constituents of treated and untreated Agave Americana fibers

The results obtained by the chemical constituents of the *Agave Americana* fibers are presented in the Table 5.

From Table 5, it is obvious that the cellulose content in sample UNS was 70.77% which increased in sample SAK with 24.27% but decreased in both the samples SACE with 1.97% and sample SB with 3.49% the reduction was higher in sample SB among the chemical treated samples. The lignin content in sample UNS was 13.59%. On chemical treatment, the sample SB exhibited an increase in lignin content with 31.3% but decreased in both the samples SACE (20.08%) and SAK (0.95%) of which the reduction was higher in sample SACE.

As for the wax content it was 0.29% in sample UNS. This was noted to reduce in samples SAK (27.58%) and SB (13.79%) but an increase with 24.13% was noted in sample SACE on chemical treatment. The ash content was observed to be 3.1% in sample UNS which reduced in all the chemical treated samples of which it was the maximum in sample SACE with 63.54% followed by the samples SB with 44.51% and SAK with 35.80%.

Alkalization partially removes the lignin, oils and wax covering the outer part of the fiber cell wall. The treatment depolymerizes the cellulose in fiber and thus opens up the short length crystallites (Mohanty, Misra, and Drzal 2001a). This trend is observed in this study in sample SAK which may assist in compatibility of fiber and matrix. The statistical analysis in one-way ANOVA done between the chemical treated and untreated samples showed that there is a significant difference at 1% level. Hence it could be concluded that the chemical treatments reduced various chemical contents namely cellulose, lignin and ash in sample SACE, Cellulose, wax and ash in sample SB and lignin, wax and ash in sample SAK. There is an impact on fibers by chemical treatments which is proved by statistical analysis.

Table 4. Spectrophotometric analysis of treated and untreated *Agave americana* fibers.

S.No.	Samples	Yellowness Index	Whiteness Index	Brightness Index
1	UNS	41.279	4.704	40.550
2	SAK	46.097	−6.330	25.428
3	SACE	46.482	−5.363	30.306
4	SB	43.401	−1.963	28.438

Table 5. Chemical constituents of treated and untreated *Agave americana* fibers.

		Chemical constituents (%)									
		Cellulose			Lignin			Wax		Ash	
S.No.	Fiber samples	Value (%)	Loss/ Gain (%)	Statistical Analyses F-value/ Significance	Value (%)	Loss/ Gain (%)	Statistical Analyses F-value/ Significance	Value (%)	Loss/ Gain (%)	Value (%)	Loss/ Gain (%)
1	UNS	70.77	-	-	13.59		-	0.29	-	3.1	-
2	SAK	87.95	24.27	21.516*	13.46	−0.95	167516.00*	0.21	−27.58	1.99	−35.80
3	SACE	69.37	−1.97		10.86	−20.08		0.36	24.13	1.13	−63.54
4	SB	68.3	−3.49		17.85	31.3		0.23	−13.79	1.72	−44.51

* -Significant at 1% level.

Moisture content, moisture regain and water absorption rate

The results obtained in moisture and water absorption behaviors of *Agave Americana* fibers are presented in Table 6.

From Table 6, it is obvious that the density of the untreated sample UNS was noted to be 1.370 g/cc. This reduced in the sample SACE to 1.236 g/cc whereas it showed a slight increase in the samples SAK and SB with 1.381 g/cc and 1.414 g/cc respectively which may be due to the densification of fiber cell walls and filling of pores by grafted molecules (Sawpan, Pickering, and Fernyhough 2011). The moisture content observed in sample UNS was 12.02% which reduced in sample SB to 11.61% but slightly increased in sample SAK to 12.17% and drastically in sample SACE to 18.04%. Moisture regain of the sample UNS was noted to be 0.60%. All the chemical treated samples showed an increase in moisture regain of which it was the highest in the sample SACE to 3.73% followed by samples SAK and SB with 2.88 and 1.83%s respectively. Statistical analysis by ANOVA also showed that there is a significant difference at 1% level in the interactions made between untreated and chemical treated samples.

The water absorption of sample UNS was 224.60% of its weight. This increased drastically in sample SB to 254.64% by its weight, but this decreased in both the samples SAK (208.86%) and SACE (176.35%) by their weights. The acetylation process decreased the diffusion of water into the fiber and fiber turned more hydrophobic which is in par with Bessadok et al. (2007). This may be due to the replacement of hydroxyl groups by hydrophobic acetyl groups. Chemical modification takes place as the acetic anhydride substitute the cell wall hydroxyl groups with acetyl groups, making the surface more hydrophobic. Statistical analysis by ANOVA also showed that there is one percent level of significance in the interactions made between untreated and chemical treated fiber samples. The main disadvantage of lignocellulosic fibers which weakens the efficiency of the composite is the inherent hydrophilic property and the nonpolar characteristics of most thermoplastics which ultimately end up in various difficulties leading to non-uniform distribution of fibers within the matrix (Bos and Donald 1999). The chemical treatments exhibited a reduction in moisture content in sample SB and water absorption in the samples SAK and SACE depicting the reduction of hydrophilic nature of the fibers.

Fiber break and elongation

The results of the fiber break and elongation of treated and untreated *Agave Americana fibers* are presented in the Table 7.

From Table 7, it is obvious that the work to break the fiber was observed to be 11.09 Nmm in sample UNS. This increased in the sample SB to 12.16 Nmm but decreased in both the samples SACE (9.30 Nmm) and SAK (9.65 Nmm).The tensile strength at break of *Agave americana* fibers is higher than the raw fibers which add the tendency of the fibers toward becoming closely packed due to the removal of chemical constituents (hemicellulose) of fibers due to the chemical treatment (SB) (Sarikanat et al. 2014). This same trend was noted in the sample SB with work to break of 12.16 Nmm. The tensile strength and elongation at break of AAFs improved on chemical treatment (Madhu et al. 2020). The statistical analysis using one

Table 6. Moisture content, moisture regain and water absorption rate.

S.No.	Fiber Samples	Density (g/cc)	Moisture content (%)	Moisture Regain		Water absorption rate	
				Value (%)	F-Value and Significance	Value (%)	F-Value and Significance
1	UNS	1.370	12.02	0.60	36572.000*	224.60	20.155*
2	SAK	1.381	12.17	2.88		208.86	
3	SACE	1.236	18.04	3.73		176.35	
4	SB	1.414	11.61	1.83		254.64	

* -Significant at 1% level.

Table 7. Fiber Break and elongation.

S.No.	Fiber samples	Work to break: Value (Nmm)	Work to break: Standard Deviation	Work to break: F Value and Significance	Elongation: Value F max %	Elongation: Standard Deviation	Maximum Breaking Force: Value Force (N)	Maximum Breaking Force: Standard Deviation
1	UNS	11.09	3.32		9.4	2.9	4.39	1.03
2	SAK	9.65	2.83	5157.333*	10.1	2.1	3.77	0.905
3	SACE	9.30	3.46		9.3	2.7	4.11	0.979
4	SB	12.16	4.13		9.2	2.0	4.97	1.25

* -Significant at 1% level.

way ANOVA showed a significant difference at 1% level in the interaction made between the untreated and chemical treated fibers. The statistical analysis showed a significant difference at 1% level. The fiber elongation was observed to be 9.4% in sample UNS. This increased in the sample SAK (10.1%) but decreased in both the samples SACE (9.3%) and SB (9.2%). The sample UNS required an average maximum breaking force of 4.39%. This increased in sample SB to 4.97 N but it reduced to 4.11 N in sample SACE and 3.77 N in sample SAK on chemical treatment.

Thermogravimetric analysis

The results of the Thermo gravimetric analysis of treated and untreated *Agave Americana* fibers are presented in the Table 8 and Figure 3.

The TGA results in Table 8 and Figure 4, depict the mass loss variation of treated and untreated *Agave americana* fibers with varying temperature. The untreated Agave fibers begin to lose weight earlier than chemical treated fibers. All the figures for TGA of the fibers show two stages for degradation in both treated and untreated fibers. Only slight mass loss was observed till 200°C and this is due to the evaporation of moisture from the fibers. The sample UNS underwent two stages of thermal degradation with 79.6% of degradation at 370°C and 99% at 440°C. In the two stage degradation of chemical treated samples the highest degradation (98.2%) peak was observed at the maximum temperature of 390°C followed by sample SACE with 97.5% at 370°C and sample SAK with 97.5% 370°C and sample SAK with 95.7% at 380°C. The thermal degradation was lesser in chemical treated samples over untreated sample UNS. Hence it could be concluded that the benzoylation treatment gave the highest thermal stability with highest temperature for maximum point of degradation among the treated fiber samples.

SEM

The surface morphology of the treated and untreated *Agave americana* fiber samples are presented in the Figure 4.

Table 8. Thermogravimetric analysis.

S.No.	Fiber Sample	Stage 1 of degradation: Temp ^{O}C	Stage 1 of degradation: Wt %	Stage 2 of degradation: Temp^{O}C	Stage 2 of degradation: Wt in %
1	UNS	370	79.6	440	99.0
2	SAK	290	20.2	380	95.7
3	SACE	310	32.0	370	97.5
4	SB	320	25.3	390	98.2

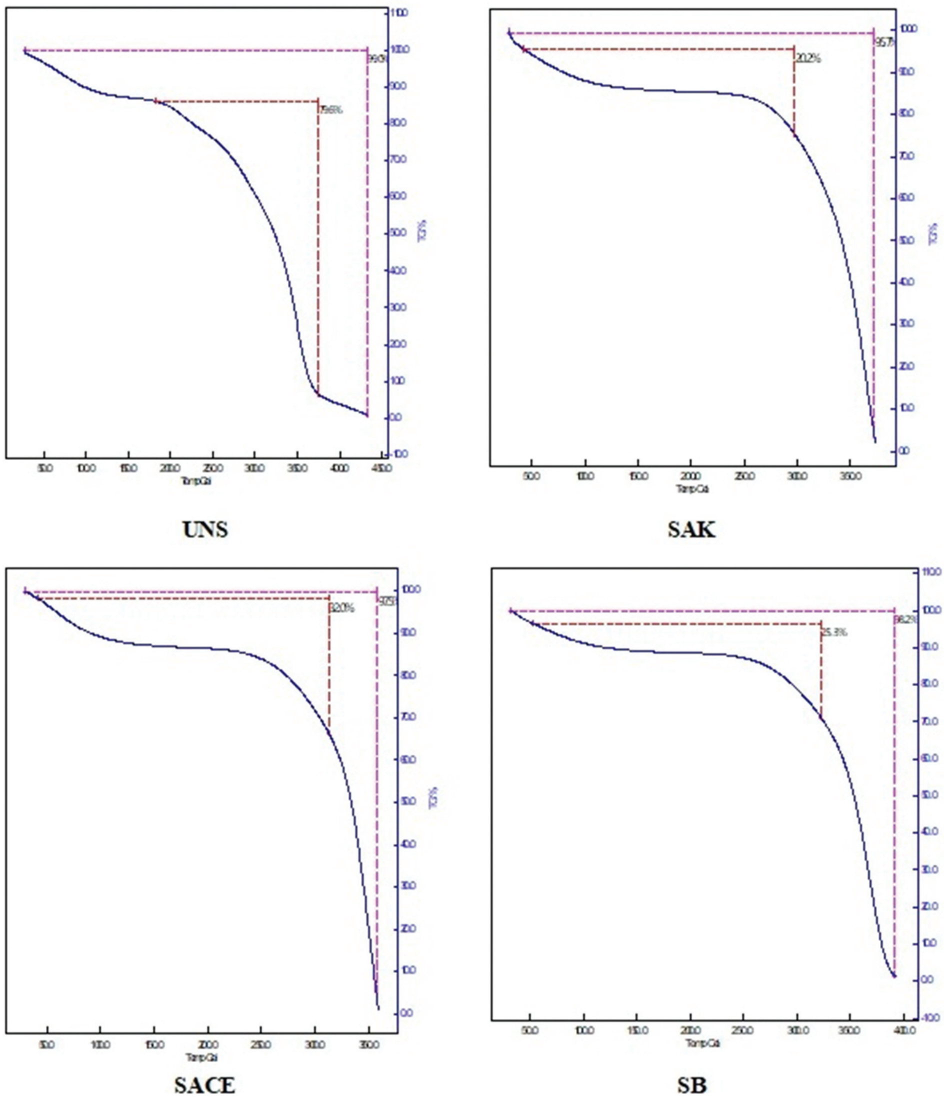

Figure 4. TGA of treated and untreated fibers.

From the Figure 5, it is obvious that the *Agave americana* fibers exhibit surface modification due to chemical treatments. Slight hairiness is noted in the samples SAK and SB depicting fibrillation which would in turn create more surface area on the fiber to interact with the matrix. On the surface of untreated *Agave americana* fiber sample (UNS) gummy materials were noted whereas a slight reduction of gummy material was observed on the surface of the treated samples namely SAK, SACE and SB. So there was a significant removal of the gummy material adhering on the surface of the treated fibers (Mohanty, Misra, and Drzal 2000). Alkalization improves the adhesive characteristics of fiber surface by removal of natural and artificial impurities, thereby producing a rough surface topography. It also leads to fibrillation, creating more available surface area on the fiber to interact with the matrix (Joseph et al. 2010a). The NaOH solution, silane and benzoyl chloride treatments altered the fiber surface to rough surface and fiber porosity was increased which may be due to the removal of hemicelluloses (Joseph et al.

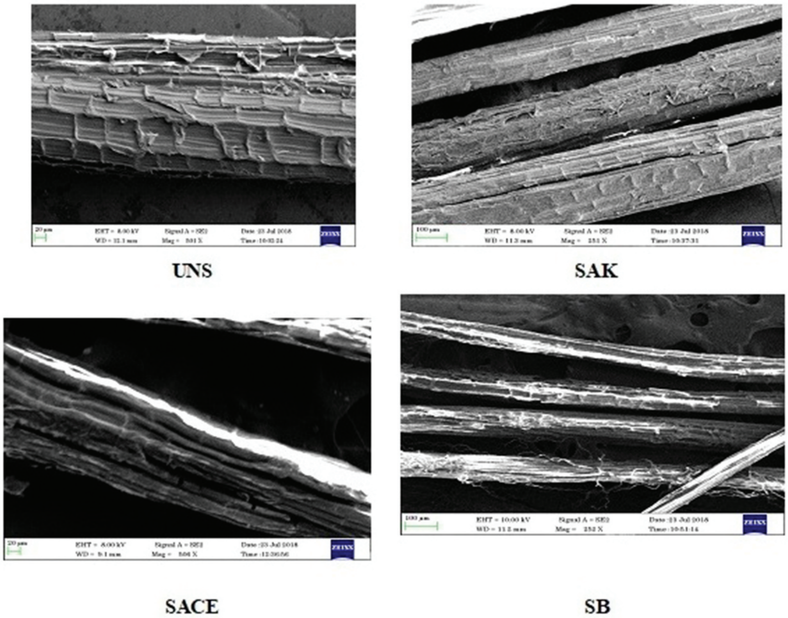

Figure 5. SEM appearance of treated and untreated fibers.

2010b). This surface modification was observed in the sample SB. These surface modifications may improve the compatibility of the reinforcement and matrix during the composite preparation.

Conclusion

In the investigation carried out in this paper, there is a novelty expressed in the results that there is an improvement in the properties of the *Agave americana* fibers which would meet the requisites of the product preparation with the composite structures. The matured leaves of the plant yield strongest fibers which could be utilized for fabrication. The purpose of the analysis made for the treated fibers was that when novel and aesthetic products are made from composites, color would play an important role. Though there was change of colour, luster, texture by chemical treatments general appearance was good by benzoylation. The brightness index was the highest on acetylation among the three chemical treated fiber samples. The chemical treatments reduced the cellulose content in samples on acetylation (1.97%) and benzoylation (3.49%) whereas lignin was reduced on alkalization (13.46%). The chemical treatment namely benzoylation reduced the moisture content (11.61%) and the alkali treatment (208.86% of its weight) and acetylation (176.35% of its weight) reduced the property of water absorption depicting the reduction of hydrophilic nature of the fibers. The work to break was improved drastically with 12.16 Nmm on benzoylation depicting the increase in strength of the fiber. Thermal stability of fibers was observed to be the highest by benzoylation among the three chemical treatments. Surface morphological analysis showed that there was modification by all the chemical treatments. Though each chemical treatment has influenced in modifying the property of the *Agave americana* fibers, the strength has been improved by benzoylation which is considered as the essential property for the raw material to be utilized for composite manufacturing. The enhancement of the fiber properties done by each chemical has been assessed which is the novelty of the study as it has brought better understanding about the selected chemicals on *Agave americana* fibers. This may be a strong route for many more innovations on fiber treatments and which could be considered for various future applications according to the suitability and requirement.

Highlights

- As futuristic approach an attempt has been made in this study to improve the compatibility between reinforcement and polymer matrix in composites by various chemical treatments.
- The general appearance of the *Agave Americana* fibers which underwent benzoylation was rated to be good in visual evaluation.
- Thermal stability improved in all the chemical treated fiber samples.
- Morphological analysis exhibited the removal of gummy substances and had improved roughness on the surface of fibers.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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ORIGINAL ARTICLE

Effect of chemical treatment on surface modification of *Abutilon indicum* fibres for composites applications

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Abstract

As people become more aware of the environmental damage caused by synthetic materials, ecofriendly materials are being developed. Natural fibres are increasingly being used in a variety of technical textile applications. The natural fibres are strong, inexpensive, and renewable that aid in the various manufacture of lightweight composites. The lignocellulosic fibres have their own advantages and disadvantages in terms of hydrophilic or hydrophobic nature and thermal behaviour while applying for various purposes in technical fields. To overcome the demerits of natural fibres, these fibres need to be treated using physical or biological or chemical methods for modifying the surface morphology, thermal degradation and moisture or water absorption properties which would result in better composite products. This study focuses on altering the salient properties of *Abutilon indicum* fibres using various chemicals for bringing potential natural fibre-reinforced composites. The chemical treatments have resulted in modification of surface morphology, chemical constituents and thermal properties of the abutilon fibres, which would result in production of better composite structures in future.

Keywords *Abutilon indicum* · Chemical treatment · Lignocellulosic fibres · Natural fibres · Surface modification · Surface morphology

1 Introduction

Natural fibres play an essential role in rising "green" economy as their manufacturing process are energy efficient, renewable and also reduce carbon release. Increased environmental awareness, societal interest, new environmental rules and unsustainable petroleum consumption prompted consideration of environmentally friendly materials [1]. Lignocellulosic fibres are advantageous for their properties such as low cost, renewable, low density, nonabrasive, consumes low energy consumption, high strength, elasticity modulus and biodegradability. These are also ecofriendly and give no residue when incinerated. These have good thermal conductivity which also finds a possibility for alternatively glass or carbon fibres in composite manufacturing [2]. In the Malvaceae family, there are 150 *Abutilon indicum* species, some of which are used as decorative plants. It is also known by a variety of names in other parts of the world, including Perum Tutti, Paniyara Hutti, Thuthi in Tamil, Petari, Jhapi in Bengali, Vellula in Malayalam, Tutti in Kanada, Kanghi in Hindi and Abutilon the Indian Mallow [3]. The plant grows to a height of 3–5 feet, and the stem is yellow in colour and 0.3–0.9 cm in diameter, with a relative density of about 1.8 cm^{-3} and soft, flexible fibres. It can be found in a variety of locations, including shady wastelands that can adapt to a variety of climatic conditions. Cellulose makes up 78.22% of abutilon fibres, whereas lignin makes up 6.14% and wax makes up 0.47%. These plants have medicinal properties as stem, leaf and root and so these find their utilisation in siddha to cure diseases. The flowers of the plant are in yellow, orange and purple colour [4]. Natural fibres hydrophilic nature causes them to absorb a greater amount of moisture, rendering them incompatible with the polymer matrix [5].

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This problem of higher moisture sensitivity occurs of hydrophilicity of fibres tend to absorbs moisture from the environment leading to stress of strain dimensional instability of the composite structure. Surface modification of natural fibres, particularly bast (bark) fibres such as flax, hemp, jute and henequen, which are used as reinforcement for composites in recent years, has become an important aspect to avoid such difficulties [6]. Natural fibres are extremely polar due to the presence of hydroxyl groups, which can easily establish hydrogen bonds with interacting resin matrices. However, lignin and waxy compounds coat the fibres, preventing the hydroxyl groups from interacting with polar matrices. Plant fibres must be subjected to physical or chemical treatment to change the surface and structure in order to produce reactive hydroxyl groups and make the surface rough for adhesion with polymeric materials [7]. Chemical changes of fibres, such as acetylation, methylation, cyanoethylation, benzoylation, permanganate treatment and acrylation, can lower the moisture absorbed by the fibres [8]. Plasticization of cellulosic fibres is caused via a well-known esterification technique called acetylation of natural fibres [9]. Natural fibre chemical components differ from one fibre to the next. Because cellulosic fibres are hydrophilic, they absorb moisture [10]. The challenge with integrating natural fibres and matrices is that the fibres must be thermally stable at rather high processing temperatures [11].

2 Materials and methods

2.1 Fibre extraction

The *Abutilon indicum* fibres were extracted from the stalk portion of the plant by the process of stagnant water retting. The *A. indicum* stalks were bundled comprising of 250–300 plants and then were immersed in a concrete tank containing soft water for a period of 10 days. Later, these stalks were tapped slightly with a wooden hammer for removing the soft pulp. The pulp was removed by scrapping with a knife. The waxy epidermal tissue, sticky pectin and hemicelluloses that attach the fibre bundles to one other were removed during the retting process [12]. Then, the fibres were separated thoroughly from the pulp, washed, combed and dried in sunlight for two days (Fig. 1).

Fig. 1 *Abutilon indicum* plants

2.2 Fibre treatments

To alter the existing hydrophobic and hydrophilic properties in fibres, several surface modifications such as mercerization, latex coating, gamma irradiation, silane treatment, isocyanate treatment, acetylation and peroxide treatment have been utilised [8, 13]. Chemical treatments such as acetylation, benzoylation and alkalization were utilised to modify the surface of *Abutilon indicum* fibres.

2.2.1 Alkalization

Alkalization is one of the most successful chemical treatment procedures for improving fibre-matrix interaction, thermal stability and heat resistance [14]. Hemicelluloses and lignin are eliminated during alkalization, and the fibre surface appears rougher than untreated fibres [15]. The *A. indicum* fibres were alkalized by immersing them in a 5% aqueous NaOH solution for 24 h at room temperature. The fibres were then extensively cleaned with distilled water to eliminate any traces of chemical and neutralise [16]. It is found that the sodium hydroxide treatment improved the mechanical properties of the biocomposites significantly due to improvement in interfacial adhesion between jute fibre and unsaturated polyester resin [17].

2.2.2 Acetylation process

The *A. indicum* fibres were soaked in demineralized water for an hour, then filtered and placed in a round bottom flask with acetylating solution (250 mL toluene, 125 mL acetic anhydride and a little amount of catalyst perchloric acid) (60%). The operation lasted for 1 h and was carried out at a temperature of 60 °C. The fibres were then rinsed with distilled water until it was acid-free and air-dried [18].

2.2.3 Benzoylation treatment

Benzoylation treatment is one of the surface modification procedures that can be used to treat fibres [19]. For benzoylation, benzoyl chloride is used, which reduces the hydrophobicity of the fibre and this enhances the fibre-matrix adhesion, increasing the strength of composites. To activate the hydroxyl groups of the cellulose and lignin the *A. indicum* fibres were given this treatment where initially the fibres were pretreated with alkali and then were suspended in 10% NaOH and benzoyl chloride solution for 15 min. The isolated fibres were then soaked in ethanol for 1 h to remove the benzoyl chloride and finally was washed with water and dried in the oven at 80 °C for 24 h.

2.3 Assessment of fibres

The treated and untreated fibres were analysed for the essential properties subjectively and objectively.

2.3.1 Visual evaluation

The untreated and chemically treated *A. indicum* samples were subjected to visual evaluation by panel of judgesof numbering 50 (Fig. 2a–d). The judges evaluated the untreated *A. indicum* fibres and chemically treated fibre samples which were displayed for evaluation for the aspects namely colour, lustre, texture and general appearance individually.

2.3.2 Spectrophotometric analysis

Fibre colour is a popular characteristic in handcrafted, textile and garment products. For textiles, colour value identification is critical [20]. Using a spectrophotometer, the yellowness index, whiteness index and brightness index of untreated and treated fibre samples were evaluated objectively. The SS 5100H premier colour scan dual beam spectrophotometer was used in the research. This equipment is built with characteristics that can be found on worldwide spectros classes which combines a highly accurate discrete sensing photodiode array detector with an advanced aberration corrected concave holographic grating for dispersion reflected light. The size of the integrating sphere is 152 mm in diameter and is in accordance with CIE/ASTM criteria. Pulsed Xenon with a wavelength range of 360 to 700 nm and a 10-nm interval is used as the light source. The equipment was adjusted at a medium area view (MAV) of 5.0 mm × 10.0 mm and a temperature of 150 °C. The relative humidity was less than 90%. Because loose fibres tend to protrude into the sphere, it was difficult to measure them. This may cause reflectance measuring problems, as well as the risk of falling into the device. Placing an exact mass of fibres into a compression cell and applying a constant amount of pressure is a preferable method. This will prevent inaccuracies caused by gaps between fibres that exist under low pressure circumstances [21]. This method was used to compare the colour of untreated and treated *A. indicum* fibres in order to determine the colour objectively for novel manufacturing of products.

2.3.3 Fibre basic properties

The fibre properties namely moisture content, moisture regain, fibre weight, density and chemical constituents were analysed as per standards in SITRA laboratory. The chemical constituents namely cellulose lignin, wax and ash present in the fibres were observed by in-house method in SITRA laboratory. The tensile strength and elongation tests were performed by using Zwick Roell tensile testing machine in SITRA Laboratory as per standards at 5 mm gauge length and 50 in speed. The tensile properties of the fibres were also analysed to obtain information about the state of the fibre bundles specifically to understand the influence of treatment on fibre properties.

2.3.4 Water absorption

One of the features of natural fibres is that they absorb water from the atmosphere via hydroxyl groups that are already present in the fibres [22]. The water absorption behaviour of

(a) Untreated *Abutilon indicum* (UNAB)	(b) *Abutilon indicum*Alkalization (ABAK)	(c) *Abutilon indicum* Acetylation (ABACE)	(d) *Abutilon indicum* Benzoylation (ABB)

Fig. 2 Images of fibres

chemical *Abutilon indicum* fibres, both untreated and treated, was examined in distilled water with a pH of 7. Individual fibre samples were bundled and dried in a hot air oven at 60 °C for 24 h before being weighed in an electronic balance with four decimal places accuracy. This was submerged in water and kept in a humidity chamber at room temperature for 24 h. After that, the fibre bundles were removed from the humidity room, cleaned and weighed. The following formula was used to compute the percentage of water absorption:

$$\text{Water absorption (\%)} = W_1 - W_2/W_2 \times 100$$

where W_1 is the mass after exposure to water and the W_2 is the mass of the dry sample.

2.3.5 Thermogravimetric analysis

Thermogravimetric analysis (TGA) determines the thermal stability of natural fibres throughout the heating process based on mass change; as a result, it is critical to understand the significant thermal stability of natural fibres at high temperatures of 220 °C for at least a few minutes [23]. Using the EXSTAR/6300, TGA was used to determine the thermal stability of untreated and treated *A. indicum* fibres. Fibre samples weighing 1 mg were placed in an alumina pan and heated between 50 and 450 °C in a nitrogen gas atmosphere. Thermogravimetric analysis was used to investigate the fibre's thermal resilience and weight loss when exposed to higher temperatures. We acquired and recorded the TGA curves of untreated and treated *A. indicum* fibre samples.

2.3.6 SEM

The SEM study of natural fibres was carried out using TESCAN-MIRA3 XMU scanning electron microscope for obtain images of the fibre surface and to evaluate the effect of chemical treatments on the fibre morphology.

3 Results and discussion

3.1 Visual evaluation

From visual evaluation, it is clear that the all treated of *A. indicum* fibres turned yellow colour and the brightness also reduced drastically on chemical treatment. The fibres were smooth and soft to touch, which depicts the removal of the adhering materials from fibre surface. From Table 1, it is clear that the cellulose content of UNAB was 67.95%, which decreased in the samples ABB, ABAK and ABACE to 65.03%, 64.3% and 62.9% respectively among the chemical-treated samples. The lignin content of the sample UNAB was observed to be 24.17%, which reduced in the

Table 1 Chemical constituents of treated and untreated *Abutilon indicum* fibres

S. no	Fibre samples	Chemical constituents (%)									
		Cellulose			Lignin			Wax		Ash	
		Value (%)	Loss/gain (%)	Statistical analysis F-value significance	Value (%)	Loss/gain (%)	Statistical Analysis F-value significance	Value (%)	Loss/gain (%)	Value(%)	Loss/gain (%)
1	UNAB	67.95	-	1794.759*	24.17	-		0.18	-	2.2	-
2	ABAK	64.3	5.37		22.81	5.62	29,959.33*	0.11	38.8	3.11	41.36
3	ABACE	62.9	7.43		25.17	−4.13		0.29	61	0.52	76.36
4	ABB	65.03	4.29		22.55	6.70		0.17	5.55	1.48	32.72

*1% significant level

samples ABAK and ABB with 22.81% and 22.55%, but an increase was noted in sample ABACE with 25.17%. The wax content of the sample UNAB was noted to be 0.18%, and this reduced in samples ABB with 0.17% and ABAK with 0.11%, whereas a slight increase was noted in sample ABACE with 0.29%.

From Table 2, it is clear that the moisture regain of the fibre sample UNAB was 1.41%. This was reduced in samples ABAK (1.01%) and drastically increased in samples ABB (4.03%) and ABACE (3.9%). This decrease in moisture regain by alkylation process may be due to the increase in the hydrophobic property of the fibre. But byacetylation and benzoylation the hydrophilic property has been induced to the *A. indicum* fibre samples. Moisture content of the sample UNAB was noted to be 12.10% which reduced in samples ABAK to 11.6%, followed ABACE to 10.99% and ABB to 10.56%.The density of samples UNAB was observed to be 1.45 g/cc, which decreased in samples ABB to 1.324 g/cc, ABACE to 1.372 g/cc and no change in density was noted in sample ABAK same density in sample (1.45 g/cc). Hence, it could be concluded that the maximum delignification was observed in sample ABB. The wax content reduced in sample ABAK and ash content substantially reduced in sample ABACE. As far as moisture content is concerned, a substantial reduction was noted in sample ABB. Density was observed to have a substantial decrease in sample ABB. The statistical study of one-way ANOVA performed for the interaction between the chemical treated and untreated samples for cellulose and lignin contents showed a significance difference at 1% level.

3.2 Spectrophotometric analysis

Whiteness is a colour quality characterised by strong luminosity and the absence of any hue. Yellowness index measurements are commonly used to investigate white colour degradation caused by raw materials, processing or after service exposure. The measurement of light reflectance of a certain wavelength of blue light is known as brightness [24].

From Fig. 3, it is obvious that the yellowness index increased drastically in sample ABAK (55.562) followed by the samples ABACE (50.133) and ABB (50.069) over

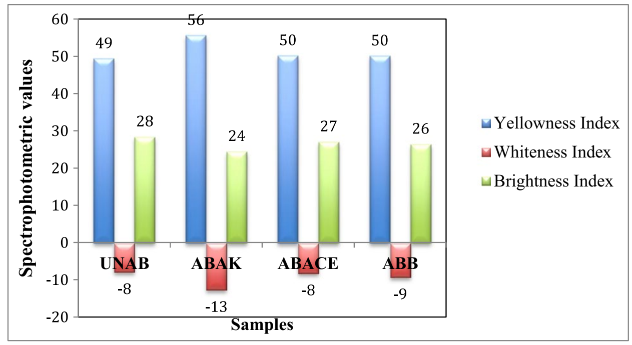

Fig. 3 Spectrophotometric analysis

the original sample UNAB (49.317). The reduction in whiteness index and brightness was observed in all the chemical-treated samples. The reduction in brightness was observed in all the treated samples over the untreated sample. The brightness index was the highest in sample ABACE (27.014) followed by samples ABB (26.368) and ABAK (24.403), which were lesser than the untreated sample UNAB.

As a result, the sample ABAK was found to have the highest yellowness index and the lowest brightness index. The chemical treatments increase the yellowness index leading to decrease in brightness of fibre samples [25]. This tendency of *A. indicum* fibres may assist in adding colour depth to the fibres by chemical treatments.

3.3 Water absorption

From Fig. 4, it is clear that the sample UNAB hold the water at 178% by its weight. This absorption property improved in the sample ABAK to 258% followed by ABB to 188%, but this reduced in the sample ABACE to 169% in the chemical-treated samples. This may be due to the removal of hydrophobic lignin from the fibre structure due to chemical treatment in both the samples ABAK and ABB which is in par [26]. Benzoylation reduces the hydrophobicity of the fibre [19], which is also proved in this study as the sample ABB exhibits higher water absorption than sample UNAB. Acetylation modifies the surface of natural fibres, making it more hydrophobic than untreated as the chemical reacts with the

Table 2 Moisture and density aspects of treated and untreated *Abutilon indicum* fibres

S. no	Fibre samples	Moisture regain	Statistical analyses	Moisture content		Density g/cc
		Value (%)	*F*-value significant level	Value (%)	Loss/gain (%)	
1	UNAB	1.41		12.10	-	1.45
2	ABAK	1.01	8582.026*	11.6	4.13	1.45
3	ABACE	3.9		10.99	9.17	1.372
4	ABB	4.03		10.56	12.72	1.324

*1% significant level

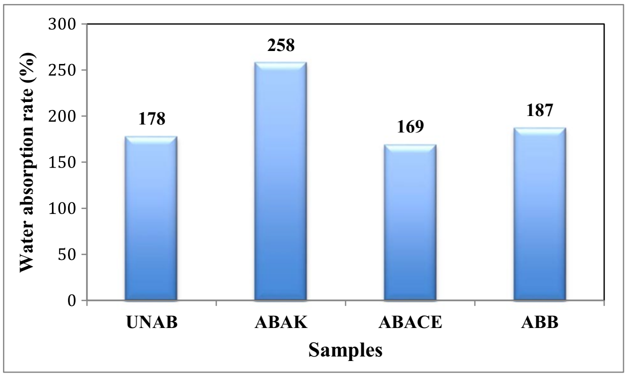

Fig. 4 Water absorption of treated and untreated *Abutilon indicum*

hydroxyl groups (OH) of the fibre [27], which is exhibited in the sample ABACE. The one-way ANOVA study performed comparing the samples to determine the influence of cellulose and lignin content variation on water absorption found a significant difference at the 1% level.

3.4 Tensile strength and elongation

From Table 2, it is clear that the breaking strength was 4.02 in sample UNAB. This improved in sample ABAK with 6.01 Nmm, whereas it decreased in both the samples ABACE 3.86 Nmm and ABB 3.41Nmm. Breaking elongation was observed to be 1.17% in UNAB which increased in both the samples ABAK (1.26%) and ABACE (1.20%) but decreased in sample ABB with 1.1%. The force required for the fibre breakage was observed to be 1.09 N, whereas UNAB required 1.88 N. The sample ABACE required 1.09 N required same as UNAB and sample ABB only 0.97 N. So single fibre strength exhibits an improvement in strength in samples ABAK (6.01%) over the sample UNAB (4.02%) which is in par with the findings of Vardhini [27] that the strength increases due to alkali treatment. The alkali treatment showed an increase in strength of 6.01% in sample ABAK over sample UNAB. This increase in strength due to alkali treatment, which has reduced the non-cellulosic content from the fibres. The standard deviation of the breaking strength, breaking elongation and force of thirty readings is presented in the Table. The ANOVA carried out between the untreated and treated fibre samples is noted to have a significant difference at 1% level.

3.5 Thermogravimetric analysis

The thermogravimetric analysis results are presented in Table 3.

Table 3 reveals that treated fibres are more thermally stable than untreated fibres. Cellulose has higher heat stability. This may be the reason for sample ABAK to have higher stability which has 64.3% of cellulose content (Table 1) than the sample ABACE which had only 62.9% cellulose content (Table 1). Though the sample ABB had 65.03% cellulose, it exhibited lesser thermal stability than sample ABAK. Thermal stability of amorphous components such as hemicelluloses, pectin and lignin is limited, and they disintegrate at temperatures below 500 °C. Table 4

The decreasing weight of fibre material as a function of rising temperature was represented in Fig. 5 panels a to d. The first step of heat degradation for untreated *Abutilon indicum* began at 390 °C, with a weight loss of over 1.7% due to the release of moisture content from the fibre. The TGA curve remains reasonably flat until the temperature approaches 450 °C, indicating that sample ABAK

Table 3 Tensile strength and elongation of treated and untreated *Abutilon indicum fibres*

S. no	Fibre samples	Breaking strength			Breaking elongation		Force	
		Values (*N*)	Standard deviation	*F*-value and significance	Values (%)	Standard deviation	Values (Nmm)	Standard deviation
1	UNAB	4.02	1.5	26,571.333*	1.17	0.23	1.09	0.52
2	ABAK	6.01	2.2		1.26	0.21	1.88	0.91
3	ABACE	3.86	1.51		1.20	0.3	1.09	0.63
4	ABB	3.41	1.44		1.10	0.3	0.97	0.56

Table 4 TGA of treated and untreated *Abutilon indicum* fibres

S. no	Fibre sample	Stage 1 of degradation		Stage 2 of degradation		Stage 3 of degradation	
		Temp °C	Wt (%)	Temp °C	Wt (%)	Temp °C	Wt (%)
1	UNAB	390	101.7	-	-	-	-
2	ABAK	280	19.5	430	69.1	450	94.6
3	ABACE	340	36.5	450	95.3	-	-
4	ABB	325	35.6	375	97.3	-	-

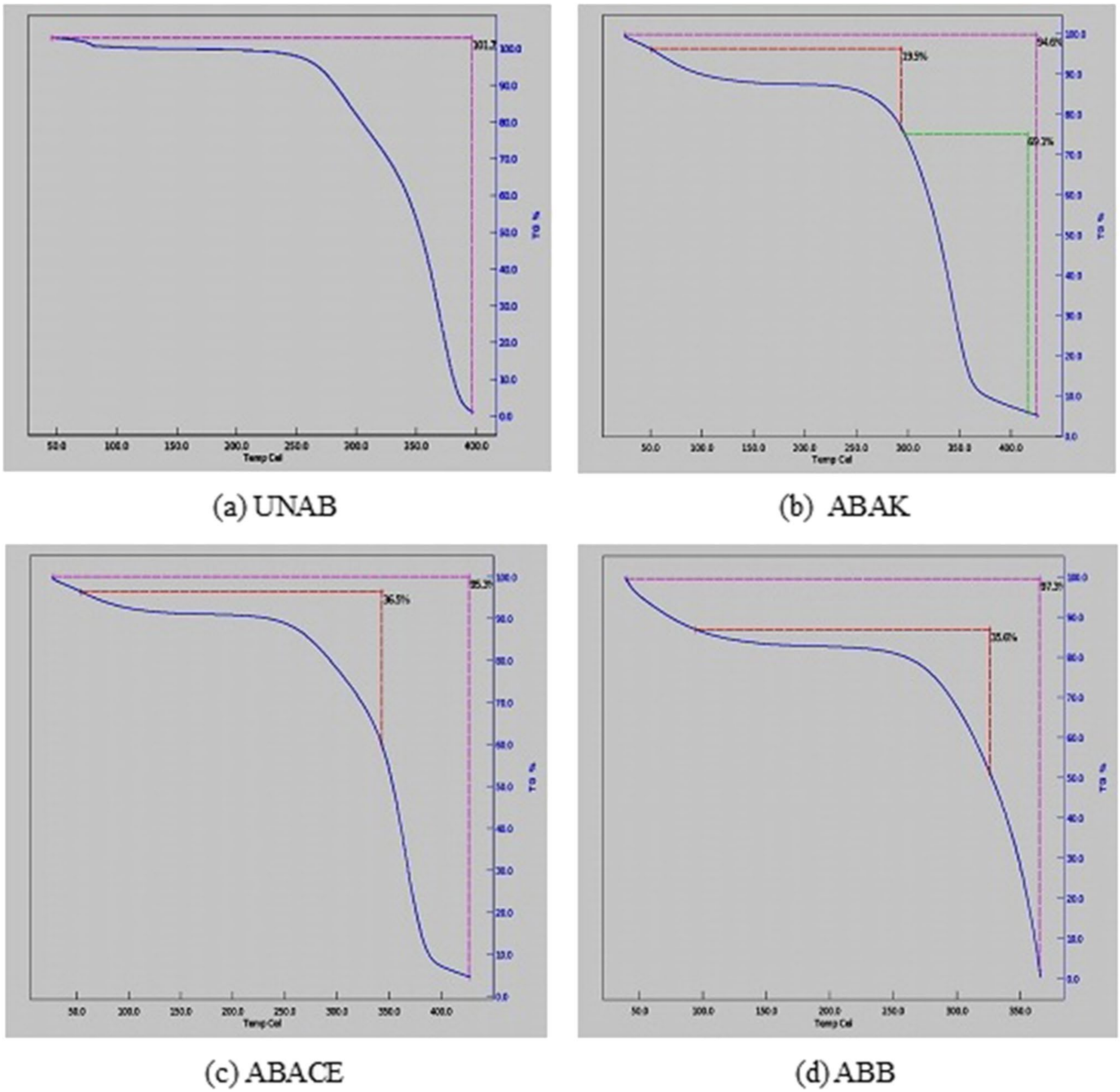

Fig. 5 TGA of treated and untreated fibres

decomposes slowly. Thermal depolymerization of hemicelluloses and glycosidic links in cellulose cause the first phase. The second stage occurs as a result of cellulose degradation, which results in a significant char residue. The first step of thermal deterioration for alkali-treated *A. indcum* fibre occurs at 280 °C, with weight loss of 19.5%, which corresponds to the evaporation of absorbed moisture content during treatment. The second stage of degradation, which resulted in the degradation of hemicelluloses and lignin, occurred at a temperature of 430 °C, with a weight loss of 69.1%. The final step of deterioration, which happened owing to cellulose degradation, resulted in a 94.6% weight loss at 450 °C. In the case of acetylation, the first stage of degradation occurred at 340 °C, resulting in a weight loss of 36.5% due to the removal of moisture content from the fibre; the second stage of degradation occurred at 450 °C, resulting in a weight loss of 95.3% due to the decomposition of hemicelluloses and lignin; and the final stage of TGA curve remains flat. The first stage of benzoylation begins at 325 °C with a weight of 35.6, and the second stage begins at 375 °C with a weight of 97.3%. All chemically treated fibres outperformed *Abutilon indicum* fibres in terms of heat stability.

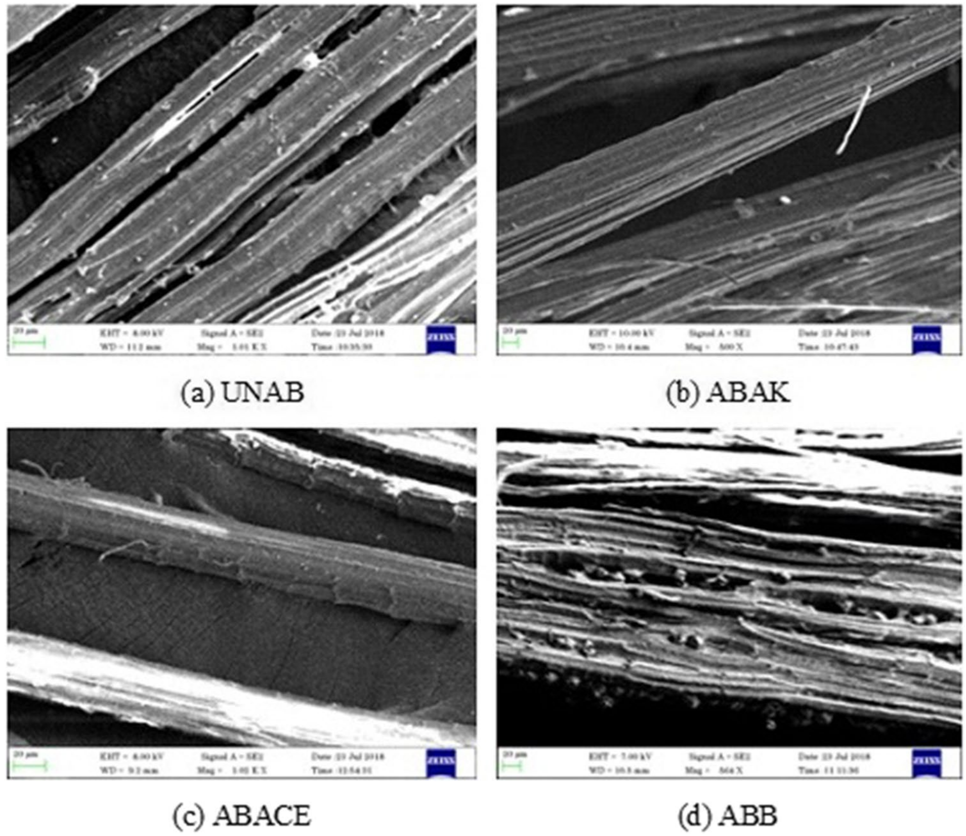

Fig. 6 SEM appearance of treated and untreated fibres

3.6 SEM

The SEM characterisation reveals a significant change in the morphology of fibres after treatments as depicted in Fig. panels 6a to d. The fibre is held together by the substances such as lignin and pectin. This is well observed in the sample UNAB. On various chemical treatments, the fibres are noted to have splits on the surface which may be due to the roughness formed on the surface of the fibres. The removal of lignin and celluloses from the surface of fibres is thought to be the cause of this morphological alteration. Thus, the morphological modification is also carried out by the chemical treatment in *Abutilon indicum* fibres.

4 Conclusion

The alkalization treatment of *Abutilon indicum* fibres reduced the hydrophilic property, whereas acetylation and benzoyl treatments have increased the hydrophilic property in the fibres, which is evident by the results obtained in moisture content and regain of the fibres. The improvement in the hydrophilic property may assist in greater uptake of matrix by the reinforcement, which would lead to manufacture of stronger composite structures. The reduction in moisture content of the treated fibres with 12.72%, 9.71% and 4.13% in samples ABB, ABACE and ABAK over untreated sample will increase the matrix and reinforcement compatibility reducing the voids in the composite structure. This prepared composite product would find their application in moisture prone areas. The chemical constituents namely cellulose and ash show highest reduction in acetylation process with 7.43% and 76.36% respectively among the three chemical treatments. Delignification was the highest by benzoyl treatment with 6.70% which has reduced the hydrophobicity of the fibre and is proved in water absorption treatments and wax removal was the maximum in alkali treatment. The chemical treatments have reduced the chemical constituents in the fibres, which is also expressed in the density test. Among the chemical treatments, the alkali treatment had improved the strength and elongation of the *Abutilon indicum* fibres to 6.01 N, which may give a better strength to the composite end product. The thermal stability also has been observed in all the chemical treated fibre samples. The morphological modification is observed through the SEM study also. The surface modification of *A. indicum* fibres has been successfully carried out by various chemical treatments namely alkalization, acetylation and benzoylation of which the strength enhancement was obviously noted in alkalization. Based on the end product to be manufactured, the appropriate chemical treatment could be selected for *A. indicum* fibres.

Author contributions ST initiated the study and designed the experiment. RS and LA co-designed the experiment to chemical Treatment on surface modification of *Abutilon indicum* fibres for composites applications. CP drafted the manuscript. ST, RS, LA and CP provided key comments for manuscript writing. All authors read and approved the final manuscript.

Declarations

Conflict of interest The authors declare no competing interests.

Consent for publication All authors agree to the publishing of the paper.

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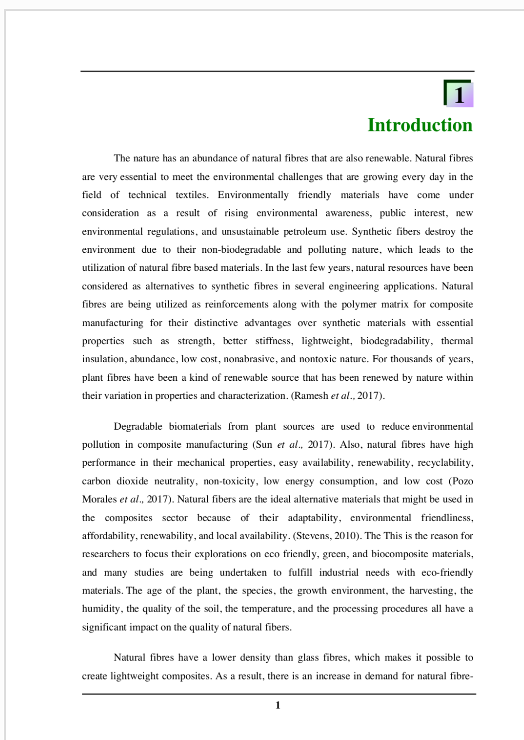

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Introduction

The nature has an abundance of natural fibres that are also renewable. Natural fibres are very essential to meet the environmental challenges that are growing every day in the field of technical textiles. Environmentally friendly materials have come under consideration as a result of rising environmental awareness, public interest, new environmental regulations, and unsustainable petroleum use. Synthetic fibers destroy the environment due to their non-biodegradable and polluting nature, which leads to the utilization of natural fibre based materials. In the last few years, natural resources have been considered as alternatives to synthetic fibres in several engineering applications. Natural fibres are being utilized as reinforcements along with the polymer matrix for composite manufacturing for their distinctive advantages over synthetic materials with essential properties such as strength, better stiffness, lightweight, biodegradability, thermal insulation, abundance, low cost, nonabrasive, and nontoxic nature. For thousands of years, plant fibres have been a kind of renewable source that has been renewed by nature within their variation in properties and characterization. (Ramesh *et al*., 2017).

Degradable biomaterials from plant sources are used to reduce environmental pollution in composite manufacturing (Sun *et al*., 2017). Also, natural fibres have high performance in their mechanical properties, easy availability, renewability, recyclability, carbon dioxide neutrality, non-toxicity, low energy consumption, and low cost (Pozo Morales *et al*., 2017). Natural fibers are the ideal alternative materials that might be used in the composites sector because of their adaptability, environmental friendliness, affordability, renewability, and local availability. (Stevens, 2010). The This is the reason for researchers to focus their explorations on eco friendly, green, and biocomposite materials, and many studies are being undertaken to fulfill industrial needs with eco-friendly materials. The age of the plant, the species, the growth environment, the harvesting, the humidity, the quality of the soil, the temperature, and the processing procedures all have a significant impact on the quality of natural fibers.

Natural fibres have a lower density than glass fibres, which makes it possible to create lightweight composites. As a result, there is an increase in demand for natural fibre-

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