MORPHOLOGY, FTIR AND X-RAY PATTERNS OF MERCERIZED PULVERIZED JACK BEAN (*Canavalia ensiformis*) SEED COATS

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Introduction

- The quest for diversification and extended-use of agro-materials is a key to globalized industrialization. Of paramount interest is unveiling the potentials of underutilized agro-materials coupled with the possibility of turning ‘green waste’ to useful raw materials, especially for non-food applications.

- Jack bean (*Canavalia ensiformis*) seed, white in colour and nearly oblong in shape, is one of the neglected under-utilized legumes [1]. It is a tropical climber producing long pendant green bean, which belongs to the family of the *leguminasae*. It is a native of West Indies and Central America, but is now found scattered throughout the tropics and sub-tropics [1].

Intro…

- Presently, scanty reports on food and non-food applications of jack bean exist. Its nutritional values, anti-nutritional substances and suitability as food supplements for man, animals and fish have been reported [3-5]. Hydroxypropylation and ozone-oxidation of jack bean starch have been reported [5, 6].

- The present work was borne out of curiosity to know the chemical constituents of jack bean seed coat, which, hitherto, is considered as a waste, and turn it to a useful raw material. As at the time of this research, the authors are not aware of any article on the characterization of seed coats of jack bean.

Aim and Objectives

- **isolating and pulverizing seed coats of jack beans (*Canavalia ensiformis*);**
- **modifying the pulverized seed coats via mercerization process**; and
- **characterizing both the unmodified and modified jack bean seed coat with a view to proposing its possible non-food applications**.

Materials and Methods

Materials

- Jack beans (*Canavalia ensiformis*) were freshly harvested from a farm in Auchi, Etsako-West Local Government Area, Edo State, Nigeria. All the reagents used were of analar grade.

Preparation of Pulverized Jack Bean Seed Coat

- The seed coats of jack beans were manually separated from the embryo, dried in direct sunlight for 4-6 days, followed by thorough manual removal of notable foreign materials such as dirts, broken cotyledons and immature seeds. The dry seed coats were pulverised in a Willey Mill (Scientific Equipment), sieved into a fine particle (250 μm) and the sample packaged in a transparent polythene bag prior to analysis

Materials and Methods…

Preparation of Mercerized Pulverized Jack Bean Seed Coat

- 50 g of the pulverized sample was soaked in 10 % NaOH solution for 1 h at room temperature. This was followed by drying at room temperature for 24 h, and oven-drying at 100 ^{o}C for 2 h.

$$\text{Seed Coat–OH} + NaOH \rightarrow \text{Seed Coat–O–Na} + H_2O$$

Analyses Carried Out

- pH
- Ash Content
- Moisture Content
- Iodine Absorption Number
- Fourier Transform Infra-Red (FTIR) Profiles
- X-ray Diffractometry (XRD)
- Scanning Electron Microscopy (SEM)

Results and Discussion

Table 1: Chemical Compositions of unmodified and mercerized pulverized jack bean seed coats

Sample	pH	Moisture Content (%)	Ash Content (%)	Iodine Absorption Number
Unmodified	6.48±0.10	4.76±0.01	**20.49±0.10**	**1.98±0.01**
Mercerized	**10.97±0.10**	**12.50±0.02**	18.18±0.10	1.00±0.01

Results…

Table 2: Major peak characteristics of unmodified and mercerized samples

Sample	Peak I				Peak II				Peak III			
	I	2θ	d	RI	I	2θ	d	RI	I	2θ	d	RI
Unmodified	1954	24.09	3.69	100	574	18.31	4.84	27	328	47.57	1.19	22
Mercerized	2201	24.06	3.70	100	543	18.30	4.84	25	276	47.53	1.91	13

I = Intensity (counts); 2ϑ = Bragg's angle; d = d-spacing; RI = relative intensity

Table 3: Major peak characteristics of unmodified and mercerized samples

Sample	$B_{(hkl)}$	θ ($2\theta^0$)	Crystallite Size, $D_{(hkl)}$
Unmodified	0.17	24.09	8.60
Mercerized	0.14	24.06	10.10

$B_{(hkl)}$= FWHM (Full Width Half Maximum), ϑ(corresponding Bragg's angle to FWHM);

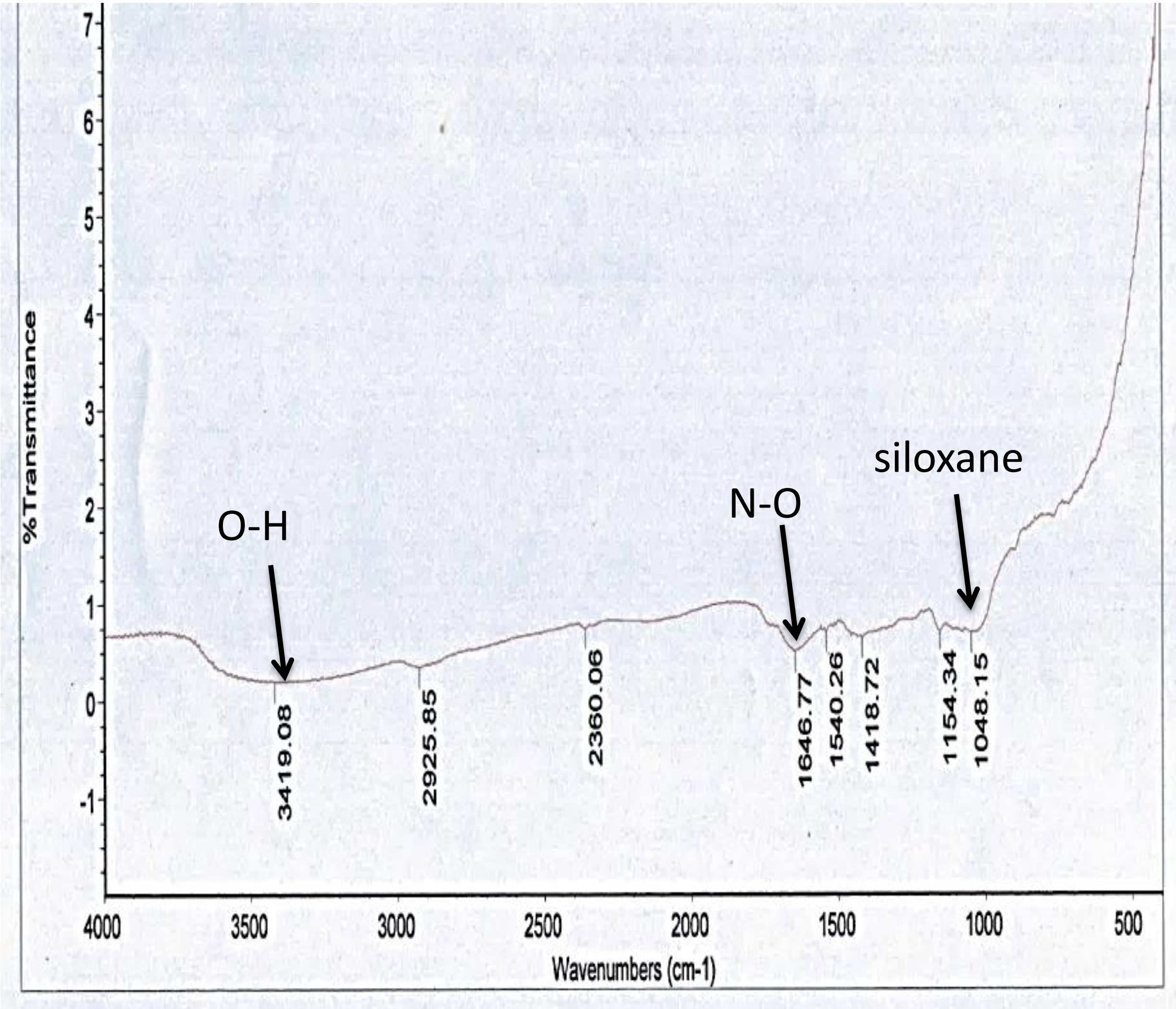
$$D_{(hkl)} = \frac{k\lambda}{B_{(hkl)}\cos\theta}$$

mixes of A- and B- polymorphs = C-type

Results…

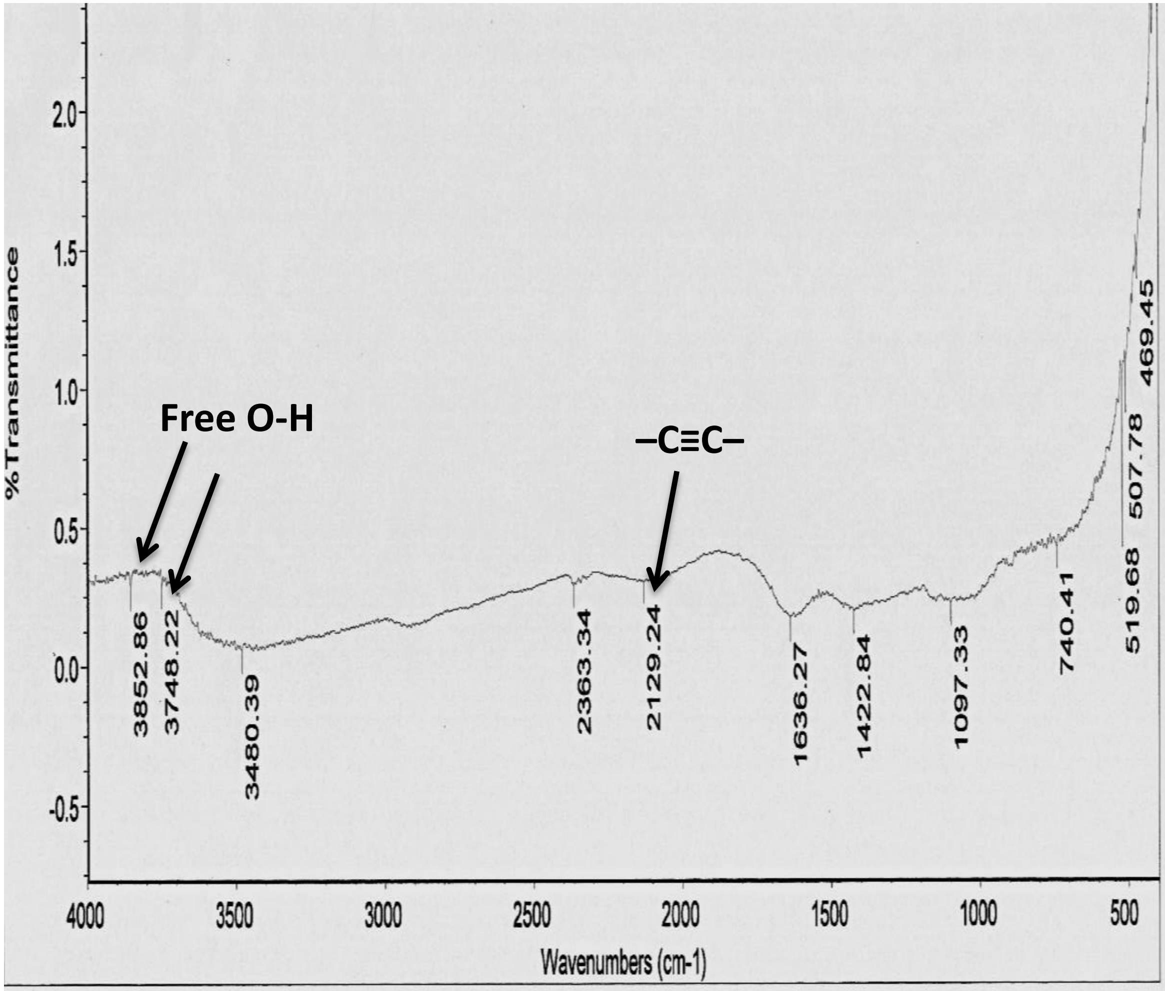
Table 4: Particle properties of unmodified and mercerized pulverized jack bean seed coats

Property	Sample	
	Unmodified	Mercerized
Circle equivalent diameter (μm)	90.20	78.30
Major axis (μm)	118.00	96.60
Minor axis (μm)	70.00	64.50
Circumference (μm)	518.00	363.00
Convex hull (μm)	375.00	293.00
Circumscribed circle diameter (μm)	147.00	114.00
Area ($μm^2$)	7.34×10^3	5.49×10^3
Volume by area ($μm^3$)	5.46×10^5	3.51×10^5
Pixel count	9309.00	7201.00
Elongation	0.375	0.328



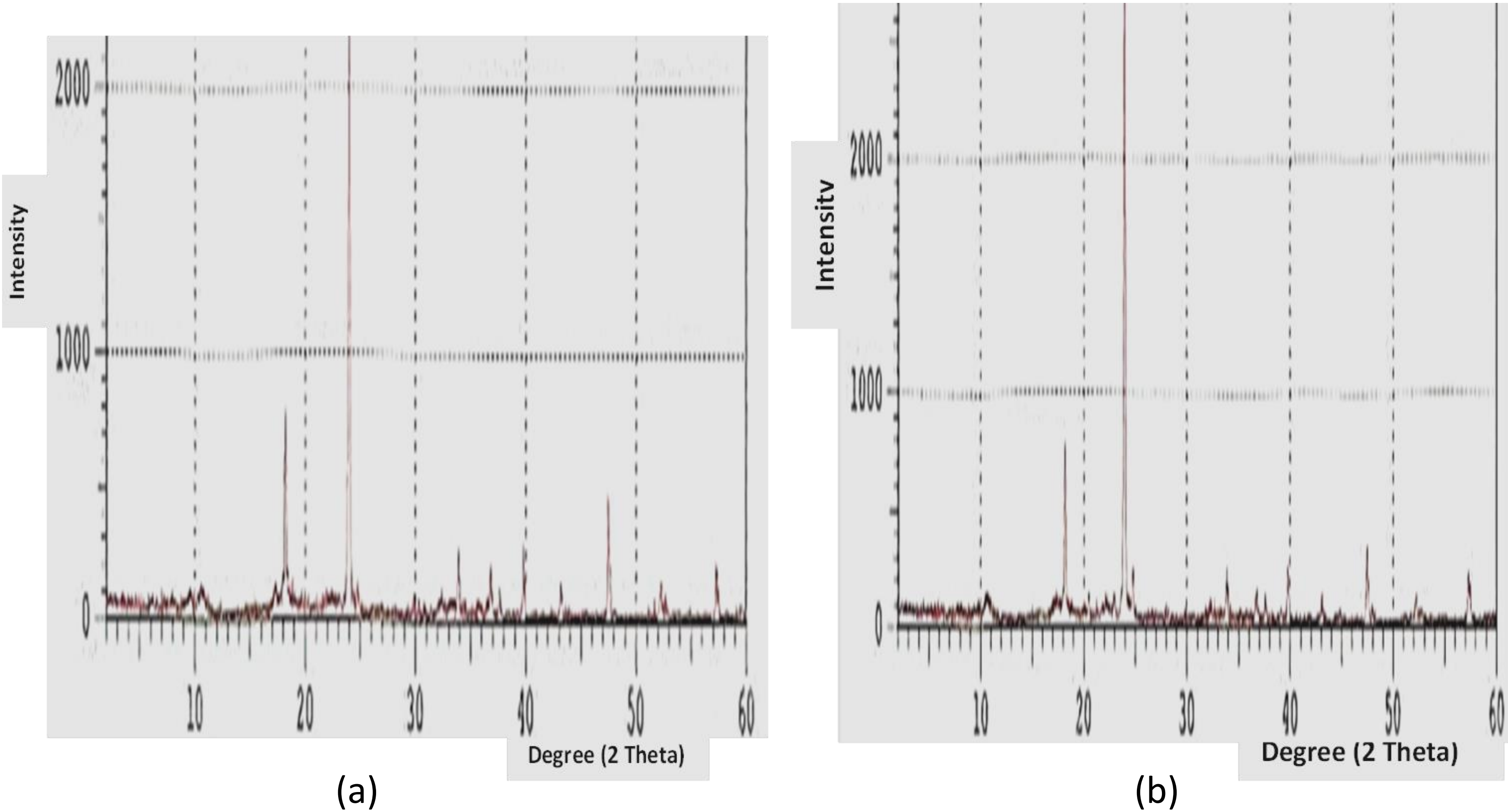
- Skeletal vibration, similar to silicon oxy-groups (of organic siloxane or silicone) and cyclohexane ring, is observed at absorption band, 1048 cm^{-1} in unmodified sample.
- Very strong N–O at 1540.26 cm^{-1}, which disappears upon modification.

FTIR spectra of unmodified pulverized jack bean seed coat



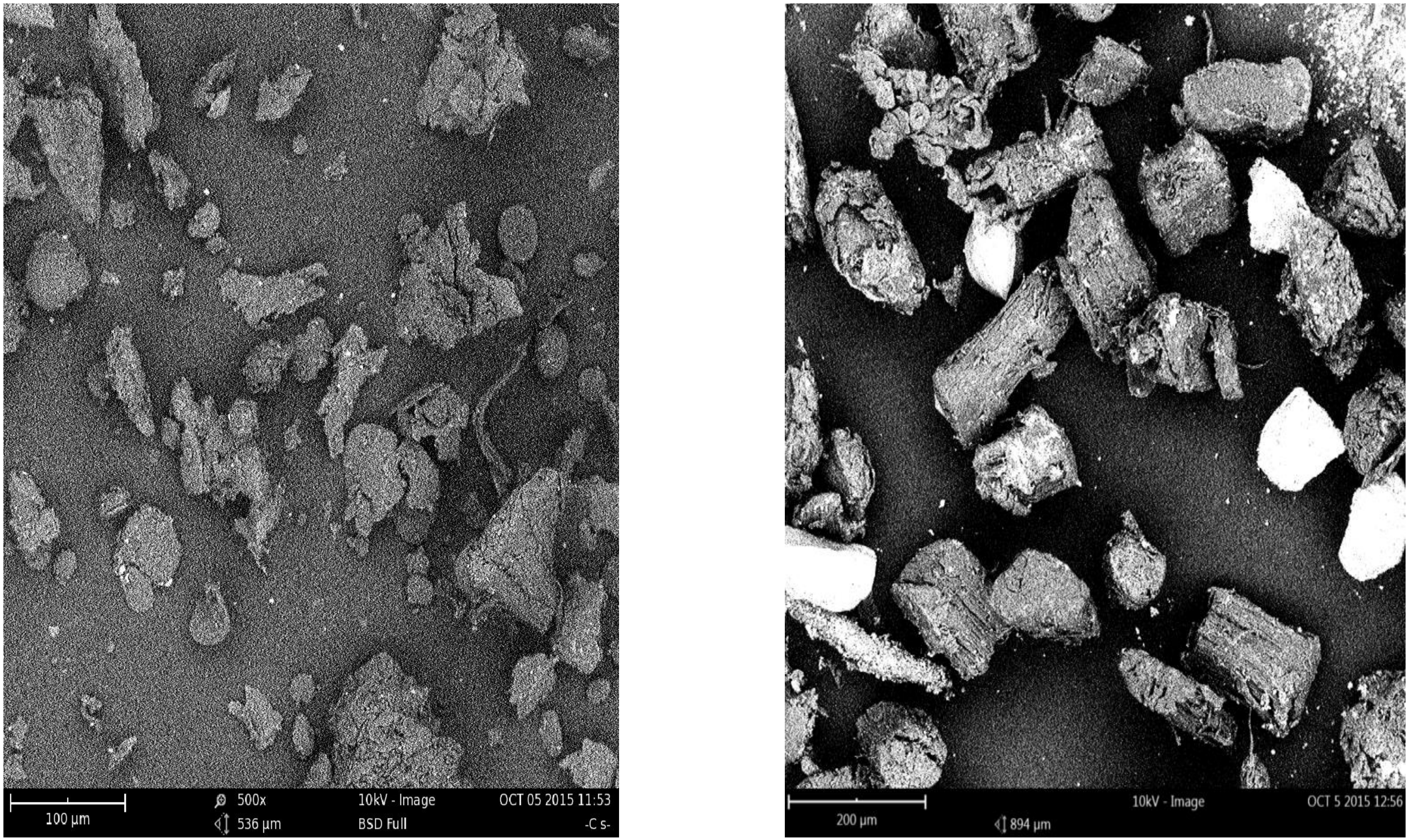
FTIR spectra of mercerized pulverized jack bean seed coat

- free hydroxyl group (O–H) stretch at 3748.22 and 3852.86 cm^{-1}
- O–H stretch of normal polymeric hydroxyl group of 3419.08 $cm^{-1}$and 3480.00 cm^{-1} for unmodified and mercerized samples respectively
- –C≡C– terminal alkynes (mono substituted) at 2129.24 cm^{-1} indicates



X-ray patterns of: (a) unmodified and (b) mercerized pulverized jack bean seed coat

mixes of A- and B- polymorphs = C-type



(a) (b)

Scanning electron micrographs of: (a) unmodified and (b) mercerized samples

Conclusion

- The pulverized jack bean seed coat has been studied with successful modification through mercerization as revealed by the FTIR spectral profiles.
- Absorption bands resembling the characteristic signal of lignocellulosics is observed in the mercerized derivative of the pulverized jack bean seed coat. The mercerized sample tends to be more reactive than the unmodified due to the unsaturated alkyne monosubstituted group (–C≡C–) observed after modification.
- The XRD profiles show that the crystallite size is more favoured by modification. Both the unmodified and the modified derivative exhibit the same XRD pattern of C-type, which is a mix of A- and B- polymorphs.

Conclusion…

- The modified pulverized jack bean seed coat is weakly acidic with comparatively high moisture content, high resistance to oxidative degradation and low surface area compared to its unmodified form.
- The SEM analysis shows irregular shapes of the unmodified granules and mixes of cylindrical and rod-like shapes for the mercerized granules. Both unmodified and mercerized pulverized jack bean seed coats can possibly serve as fillers in rubber compounding.

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Thank You!!!