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Eco-Friendly Urea-Formaldehyde Composites Based on Corn Husk Cellulose Fiber

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Abstract Cellulose fibers were extracted from dried raw corn (*Zea mays*) husk, is the second most widely traded cereal after wheat, by alkaline treatment (mercerization), followed by neutralization with acid; product of which underwent bleaching to produce pure form of cellulose. Urea-formaldehyde (UF) resin was prepared from formaldehyde and urea and its composite with the extracted cellulose fibers was prepared *via* solution casting method. Characterization of prepared samples was carried out by X-Ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), compressive strength test and water absorption test. From the results of XRD, FTIR and SEM, extraction of pure cellulose was affirmed, crystalline nature of cellulose was confirmed and crystallite size of thus obtained cellulose was also determined by XRD. From the results of FTIR, compressive strength test and water absorption test, it was concluded that addition of cellulose fiber to UF resin increases the compressive strength and water absorption of the composite which ultimately makes the composite more bio-degradable and eco-friendly.

Keywords Cellulose, FTIR, SEM, Urea-formaldehyde resin, Water absorption

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1. Introduction

Bio-based industrial residues are increasing every day. Alongside is increasing cost of their management. That is why, use of bio-based industrial residue as industrial raw material is now a growing trend among entrepreneurs [1]. Value added utilization of such residues in accordance with environmental requirements has motivated many young researchers [2,3]. Every responsible proficient global citizen is now concerned about developing sustainable and renewable polymeric material which would be able to replace petrochemical based pollution- prolonging plastic materials. Natural fiber reinforced plastics prepared using biodegradable polymer as matrix are the most environmental friendly materials [4]. One of the many efforts include, reinforcing thermosetting polymers with natural fibers for making it more bio-degradable and eco-friendly. Natural fibers when used in reinforcement of polymeric materials give enhanced mechanical and thermal stability proving them to be a better option of pure petrochemical based polymeric materials [5]. Cellulose, the most abundant biopolymer on earth, is chemically defined as a linear homopolymer composed of β-1, 4-linked glucose molecules, [6,7]. It provides excellent properties such as high mechanical properties, high strength, low thermal expansion, low density and biodegradability [6,8]. The recent demand in materials research is to develop materials which comprise excellent features such as enhanced mechanical properties and thermal stability, biodegradability, being eco-friendly, and low-cost [6]. Therefore, cellulose fibers are found to be one of those interesting co-product of bio-based industries which can be used to fill or reinforce thermosetting polymers for preparation of natural fiber composites. These materials facilitate waste management by biological processes having various applications in the economic fields where the biodegradability and renewability are preferred characteristics features [9]. Considerable contribution has been given in research using thermosets and thermoplastics as matrixes and natural fibers as reinforcements for the preparation of composites. The most commonly used thermosets are phenol formaldehyde, resorcinol formaldehyde, epoxy and urea–formaldehyde resins [10,11]. Urea-formaldehyde (UF) resin, one of the most important formaldehyde resin adhesives, is a polymeric condensation product of formaldehyde with urea [12]. However, easily available cellulose fiber used in paper and textile industries is not yet investigated as reinforcements, despite its high potential of replacing traditional glass and carbon fibers. Natural fibers are easily available, light weight, cheaper, easily separable, non-corrosive and biodegradable. Additionally, they have good thermal properties in

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comparison to traditional reinforcement materials such as glass fiber, carbon fiber etc. [5]. Keeping in mind these massive advantages of cellulose fibres, the present work is designed to fabricate corn husk (*Zea mays*) fiber as reinforced urea-formaldehyde based polymer composites and study the mechanical properties and biodegradability *via* compressive test and water absorption test respectively.

2. Materials and Methodology

2.1. Materials Required

Sodium hydroxide (97% Merk); Acetic acid (99.5% Fisher Scientific); Hydrogen peroxide (30%); Urea (99.5%); Formaldehyde (37% Fisher Scientific) and Sulphuric acid (97% Merk); were of analytical grade and used without further purification. Corn husk was obtained from local farm house, Panchkhal, Kavrepalanchowk District, Nepal.

2.2. Extraction of Cellulose From Corn Husk by Chemical Modification

Different stages involved in the extraction of cellulose from corn husk are depicted in Figure 1. According to Chirayil et al., pre-treatment with sodium hydroxide and sodium chlorite (bleaching), has removed the noncellulosic constituents resulting in fibers with high cellulose content [13]. Therefore, three simple steps of mercerization (alkali treatment), neutralization and bleaching were involved to obtain high cellulose content material from corn husk. *I. **Mercerization***; Corn *(Zea mays)* husk were sun dried until completely dehydrated and cut into small pieces of almost 2 to 3 cm and treated with 0.5 N sodium hydroxide (NaOH) solution for 3 hours at 70°C with 5% weight of cornhusk in alkali solution. This process removes lignin, hemicellulose, waxes and other surface impurities of fiber. Therefore this process is also called mercerization [1].

*II. **Neutralization***; The treated slurry was washed in water to remove dissolved substance and the coarse fibers obtained from were neutralized using 10% V/V acetic acid solution. The neutral state of coarse fiber was then dried under ambient condition inside a desiccator for 24 hours. This helped the fiber regain neutral state and also further removed surface impurities. After neutralization, it was oven dried and bleached.

*III. **Bleaching***; The dried corn fibers were bleached by using 3 g/L hydrogen peroxide at 70°C for 3 hours with 7% W/V of fibers in bleaching solution. Bleaching leads to further delignification of fiber and gives white cellulose [14]. The obtained white cellulose fibers were then washed several times by distilled water and were oven dried assuring no presence of moisture. The thus obtained fiber was then grinded in a cleaned distilled-water-washed kitchen grinder and sieved using 400 µm sieve mesh. During this process of bleaching the pH was maintained to be slightly basic. This can be done by addition of NaOH solution. The preferred pH for bleaching of natural fiber was 9 [15].

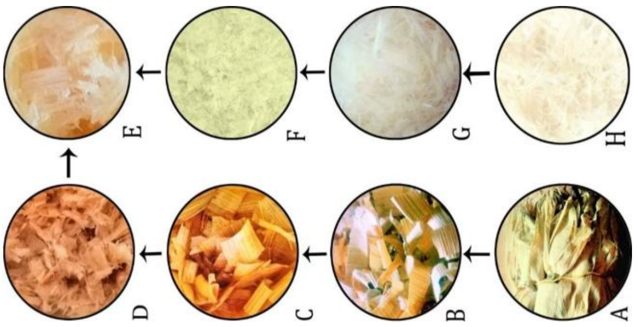
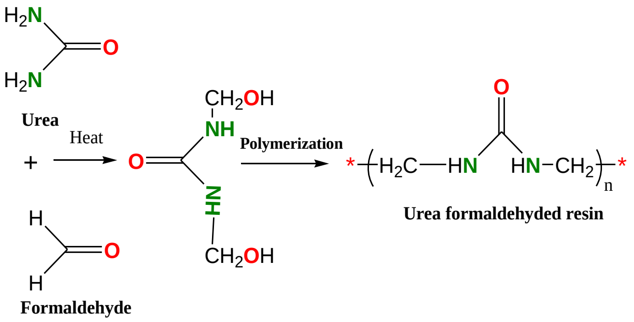


Figure 1. Extraction of cellulose from corn husk A) sun dried corn husk B) husk powder C) mercerization (alkali treatment) D) dried mercerized E) neutralized F) dried neutralized G) bleached cellulose H) dried bleached cellulose fibers

2.3. Synthesis of Urea Formaldehyde (UF) Resin

The UF resin was synthesized in laboratory (Scheme 1) by following standard protocols [16,17] with slight modification; using 1:3 (W/V) ratio of urea and formaldehyde. Formaldehyde (18 mL) in the beaker was heated on paraffin oil bath for 10 min and 6 g of urea was added to it (1 gram urea at a 1 min interval). This procedure was carried out in a magnetic stirrer for consistency in the stirring procedure. Few drops of concentrated sulphuric acid was added to the UF resin thus formed and further heated until viscous mass was obtained. Thus, obtained UF resin viscous mass was poured into plastic ice tray with grooves of dimensions (~50 cm ×30 cm ×20 cm) and was pre cured a 50°C for 24 hours and post cured at 80°C for 48 hr.



Scheme 1. Schematic route for the synthesis of urea-formaldehyde (UF) resin

2.4. Preparation of Urea Formaldehyde/Corn Cellulose Fibers (UF/CC) Composites

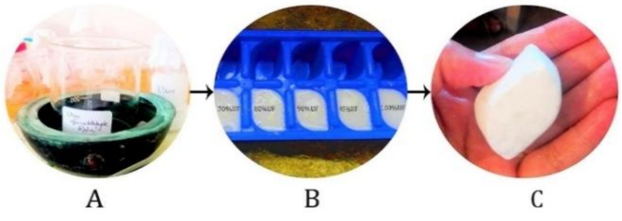


Figure 2. Preparation of urea formaldehyde/corn husk cellulose composites (UF/CC)- A) Mixture of Urea and Formaldehde (1:3), B) Different composites prepared by solution casting method, C) Cellulose based UF composite

Cellulose fibers based urea formaldehyde composites were prepared using solution casting method (Figure 2). The composites of different compositions 0/100, 5/95, 10/90, 20/80 and 30/70 (by weight) of fiber were prepared (Table 1). Thus obtained natural fiber cellulose was characterized by XRD, FTIR and SEM and its composites by FTIR, compressive strength test and water absorption test.

Table 1. Composition of Urea Formaldehyde/Cellulose Composites

S.N.	UF/Cellulose composite	Amount of UF resin	Amount of Cellulose fiber
1.	100% UF	20 mL	0.000 g
2.	95% UF	19 mL	0.333 g
3.	90% UF	18 mL	0.666 g
4.	80% UF	16 mL	1.332 g
5.	70% UF	14 mL	G

2.5. Characterization Techniques

2.5.1. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis was carried out in Central Department of Chemistry, Tribhuvan University, Kathmandu, Nepal by using IR Prestige-21 Spectrometer Shimadzu, Singapore. The spectra were collected in the spectral range of 4000-400 cm^{-1} with spectral resolution of 4 cm^{-1} in ATR mode.

2.5.2. X-Ray Diffraction (XRD)

XRD (Bruker D_2 Phase) with a monochromatic CuKα radiation source (λ=0.15406 mm) at scanning rate of 0.1 degree per step and 2θ ranging from 20 to 80 degree at Nepal academy of Science and Technology (NAST), Khumaltar, Lalitpur, Nepal. The accelerating voltage 35 KV and emission current of 30 mA were used.

The average crystallite size of the extracted cellulose was determined with the help of Debye Scherer Formula [18] (equation 1):

$$\text{Particle size (D)} = \kappa\lambda/\beta \cos\theta \qquad (1)$$

where, D = crystalline size (nm)
κ = Scherrer's constant = 0.9
λ = wavelength of X-ray used = 0.15406 nm
θ = peak position (radians)
β = Full width at half maximum (FWHM) intensity of intensity profile (in radian) [18].

2.5.3. Scanning Electron Microscopy (SEM)

Extracted cellulose fibers were characterized by SEM (FEGSEM MAIA3, HV=2kV, model 2016, Tescan, Czech Republic) to have better understanding of the surface structures and morphologies.

2.5.4. Compressive Strength Test of UF/CC Composites

The UF/CC composites were analyzed by measuring the compressive strength which was performed according to directives of American Society for testing and material (ASTM) for each sample. It was carried out by Compressive Testing Machine (CTM, capacity 500 kN, Harris & Tarris Co.) at the Central Material Testing Laboratory at Pulchowk Engineering Campus, Pulchowk, Lalitpur, Nepal. The calibration factor of the instrument was 3.7 Kg and blocks of average 15 g were tested.

2.5.5. Water Absorption Test of UF/CC Composites

Water absorption test is required to predict the biodegradability of the composites. Water absorption on the materials allows microorganisms, such as bacteria and fungi to grow and utilize cellulose as a carbon source. The results implied that the sample with higher cellulose contents exhibited better biodegradability due to the fact that biodegradation is naturally caused by the penetration of the microorganisms using water as a medium. [19,20]. Composites with same dimension were immersed in clean distill water. Weight measurements were taken at of 6 hr, 24 hr, 48 hr, 72 hr, and 144 hr of immersion initially and weekly thereafter. The amount of absorbed water was calculated using the following equation 2 [21,22]:

$$\text{Water Absorption (\%)} = \left(\frac{M_2 - M_1}{M_1}\right) \times 100 \qquad (2)$$

Where M_2 is the weight before the test and M_1 is the weight during measurement (g).

3. Results and Discussion

3.1. Fourier Transform Infrared Spectroscopy (FTIR) of Corn Husk and Extracted Cellulose

Figure 3 displays the FT-IR vibrational spectrum of wave number 4000–500 cm^{-1} of natural fiber and cellulose. It showed broad absorption bands between 3600-3000 cm^{-1} which represents the O-H stretching vibration and very strong intra and intermolecular hydrogen bonds vibration of O-H of methyl and methylene of cellulose which is more prominent in case of extracted cellulose in comparison to the raw corn husk [23]. An axial C-H stretching vibration can be seen at 2921 cm^{-1} which is also more prominent in case of cellulose. FTIR peaks at 2915-2820 cm^{-1} are by C-H stretching. The peak at 1640 cm^{-1} is representing C=O stretching of hydrocarbon [23,24]. Characteristic stretching of C-O and C-O-C pyranose ring vibration of cellulose is represented by 1157 cm^{-1} peak [4,25]. The strong absorption band at 1034 cm^{-1} is observed which is characteristic of cellulose as supported by Fan *et al.* [15].

The peak at 1030-1040 is due to C-O-C stretching [26]. The absorption peak at 892 cm^{-1} is typical structure of cellulose representing C-O-C stretching vibration for β, 1, 4-glycosidic linkage [23-28]. The FTIR of cellulose and raw corn husk preserves similar profile in each wavenumber. The intensities are found higher in case of cellulose which is also reported by Asrofi *et al* [29] in case of Water hyacinth. This result of FTIR addresses successful extraction of pure form of cellulose from corn husk, additionally showing the changes occurring in the path of extracting cellulose from corn husk.

Table 2. Frequencies of Different Vibration Mode in Fourier Transform Infrared Spectroscopy Readings of Cellulose and Raw Corn Husk

Wavenumber (cm^{-1})	Functional group	Vibration mode
3346	O-H	Stretching
2921	C-H	Asymmetric stretching
1640	C=O	Stretching
1157	C=O and C-O-C	Pyranose ring vibration
1034	C-O-C	Stretching
892	C-O-C	Stretching vibration for β, 1, 4-glycosidic linkage

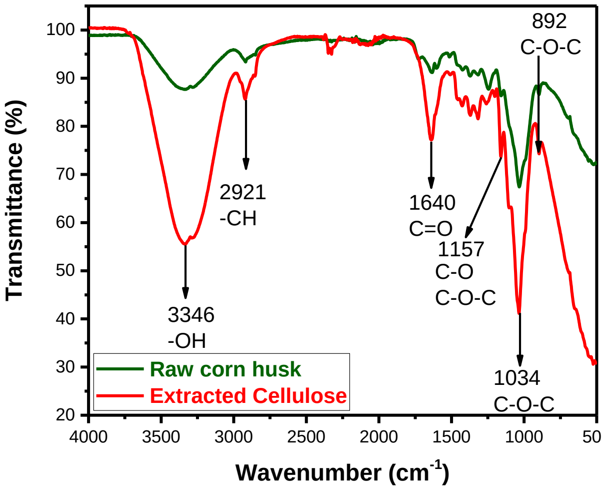


Figure 3. Fourier Transform Infrared Spectroscopy of cellulose and corn husk

3.2. X-ray Diffraction (XRD) Analysis of Corn Husk and Extracted Cellulose

Figure 4 compares the XRD patterns of raw corn husk and extracted cellulose. The average crystalline size of the extracted cellulose was determined 70 nm with the help of Debye Scherer Formula [18] (equation 1). XRD analysis of cellulose revealed the signatures characteristic of cellulose I structure, with XRD peaks located at 22.4 and 34.6° 2θ which correspond to (021) and (040) latiice plane respectively [30]. Whereas weak broad peak at 28.0° 2θ corresponds to a combination of 130, 131, 221, 227, 230 and 310 reflections [31]. The peak at 28 is very broad and weak which is affected by scattering from the paracrystalline structure and by moisture [31]. Conversely, the XRD pattern of raw corn husk appeared to be amorphous, with slight crystallinity observed at 22.0° 2θ that correspond to (021) crystallographic planes of cellulose II. On removing non-cellulosic constituents of corn husk by chemical modification, the intensity of peak become more intense and defined. Crystallinity of raw fiber is characteristically lower in comparison to that of cellulose which proves that chemical treatment successfully increased the cellulose fiber crystallinity due to removal of hemicellulose and lignin contents during the chemical treatment as also reported by Janoobi et al. [32].

Crystallinity of cellulose defined by intensified peaks confirmed extraction of pure form of cellulose from corn husk. This verifies that mercerization chemical modification technique as a very good method for easy extraction of cellulose from corn husk.

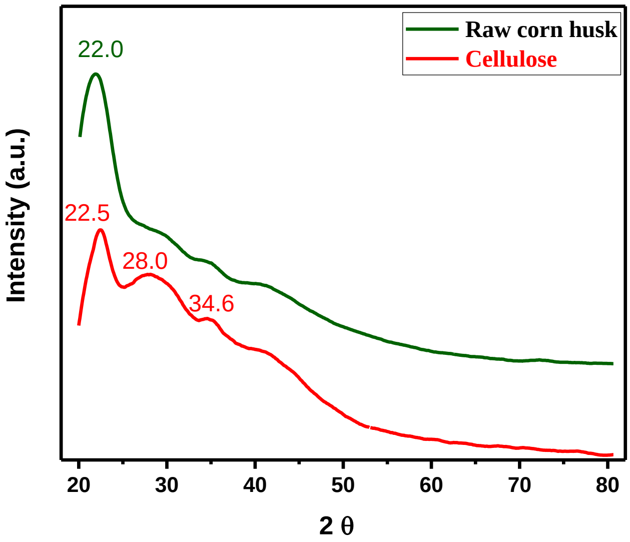


Figure 4. X-ray Powder Diffraction pattern of cellulose and corn husk

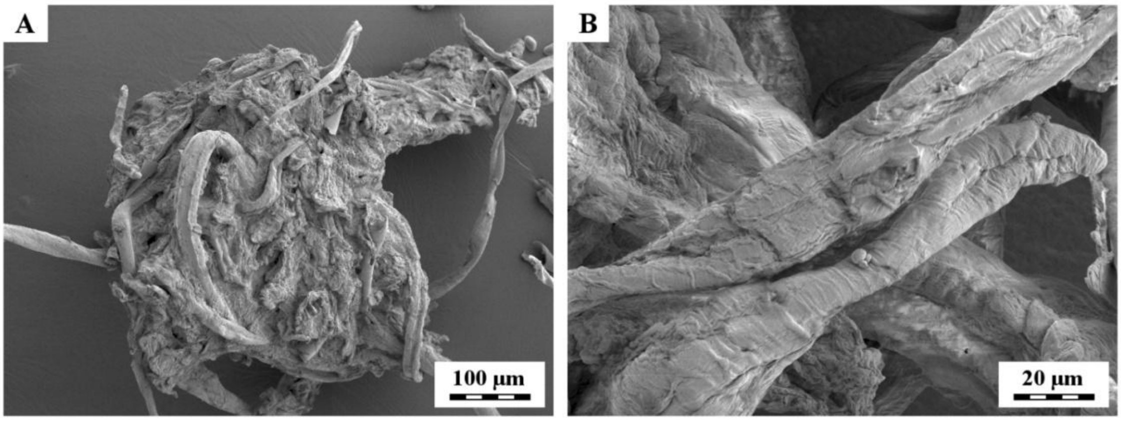


Figure 5. Scanning electron micrograph of dried cellulose fibers at (A) lower and (B) higher magnification

3.3. Scanning Electron Microscopy (SEM)

The surface morphology of the cellulose fibers was examined by scanning electron microscopy which, selected SEM micrographs are shown in Figure 5. The micrographs showed vascular texture of fibrils. The SEM results suggested that mercerization caused fibrillation and breaking of fibers to small pieces that increased surface area as previously studied by Kunusa *et al* in corncobs [33]. Petersson and Oksman [34] studied extensively microcrystalline cellulose (MCC) as reinforcement for polymers. MCC, particles of hydrolyzed cellulose consist very large amount of cellulose microcrystals together with amorphous areas. Exfoliation can be improved by swelled MCC prior to solution casting processing [34].

3.4. Characterization of Urea Formaldehyde/Corn Husk Cellulose Composites

Characterization of urea formaldehyde/cellulose composite was carried out by FTIR, compressive strength test and water absorption test to find the mechanical strength and biodegradability or eco-friendly nature of the matrix composite.

3.4.1. Fourier Transform Infrared (FTIR) Spectra of Urea Formaldehyde/Corn Husk Cellulose (UF/CC) Composite

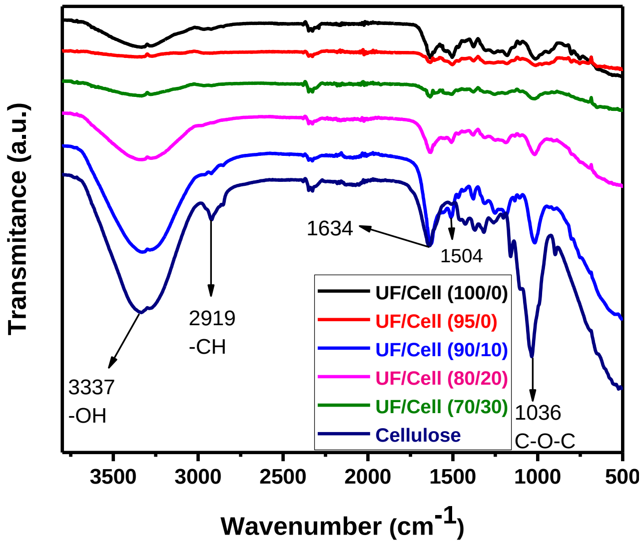


Figure 6. Fourier Transform Infrared spectra of urea formaldehyde/corn husk cellulose composites

Figure 6 present the FT-IR vibrational spectrum of wave number 4000-500 cm^{-1} of Urea formaldehyde/cellulose composite. Urea formaldehyde resin showed characteristic peaks at 3290 cm^{-1} for N-H stretching of primary amine urea, 2962 cm^{-1} for C-H stretching peak of UF, 1650 cm^{-1} for C=O stretching vibration and 771 cm^{-1} for N-H bending of secondary aliphatic amines [16,17]. The distinct absorption peak at 3337 cm^{-1} of urea formaldehyde resin is attributed to N-H stretching of primary aliphatic amines. The peaks at 3335-3340 cm^{-1} attributes O-H stretching intramolecular hydrogen bonds. FTIR peaks at 2915-2820 cm^{-1} are due to C-H stretching [16,17].

The peak at 1634 cm^{-1} is due to –C-N and –C-H overtone bending [35]. Most prominent peak at 1504 cm^{-1} represents C=O stretching peak in amide. This proves the presence of amide group in UF resin. The peak at 1263 cm^{-1} is due to C-N stretching of amide. The absorption band at 1158 cm^{-1} is C-O stretching of aliphatic ether and at 1030-1040 is due to C-O-C stretching [16,36]. The shift in peaks of C-H stretching to around 2915-2820 in composites from 2923 cm^{-1} in pure UF resin suggests some chemical interaction between matrix and reinforcement to some extent. The shifting of usual C-H stretching peak suggests that –CH_2 group played significant role in connecting fiber and matrix interface. Since there is no significant peak shift in the FTIR spectra of the constituents in the composites, it can be concluded that no strong chemical interaction or strong chemical bonding occurred at matrix-filler interface [37,38].

3.4.2. Compressive Strength Test

From the graph (Figure 7), it is seen that increase in amount of cellulose in UF resin composite increased the compressive strength which supports the work by Sharma *et al* for other similar UF resin reinforced with natural fiber [39] and Singha and Thakur for UF resin itself [40,41]. This suggests that UF resin reinforced by cellulose fibers is capable enough to to acquire good mechanical properties. The decrease in hardness in the fiber composite from pure composites attributes to fiber-matrix interfacial bond formed in the composites. This is found to improve the mechanical properties of fiber composites by making it more flexible and shock resistant [39].

3.4.3. Water Absorption Test

The samples were found to absorb water significantly on initial week and the increment of absorbing percentage became almost constant thereafter.

Table 3. Frequencies of Different Vibration Mode in FTIR Spectra of Urea Formaldehyde/Corn Husk Cellulose Composites

S. N.	Wavenumber (cm^{-1})	Functional group	Vibration mode
	3337	N-H	Stretching
	2919	C-H	Asymmetric stretching
	2634	-C-N and N-H	Overtone bending
4.	1504	-C=O	Stretching
5.	1263	-C-N	Stretching
6.	1030	C-O-C	Stretching
7.	764	N-H	O-H out of plane bending

Comparative analysis addresses more water uptake by resin with more percentage of cellulose. Figure 8 clearly states how the amount of cellulose content in UF resin makes the composite more prone to absorbing water. This is due to the –OH group in the corn cellulose structure [19,20]. This result is similar to that of an experiment conducted by Abdullah *et al.* where 25%, 50% and 75% UF resin reinforced with oil palm trunk were analyzed, which concluded that water uptake increases with increased amount of natural fiber. This work also supports the same result *i.e.* more water uptake with more proportion of natural fiber in composites [21]. Sgriccia *et al.* also revealed similar results from water absorption test for natural and glass fiber composites [42]. The pattern of water uptake in this work is found similar to that of previous works.

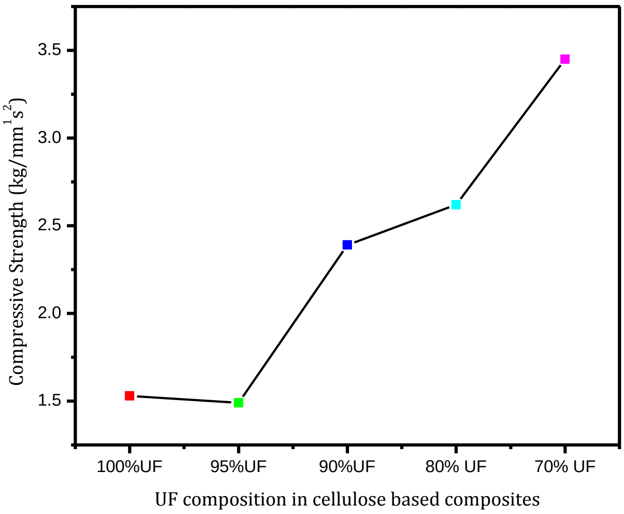


Figure 7. Compressive strength of urea formaldehyde/corn husk cellulose composites

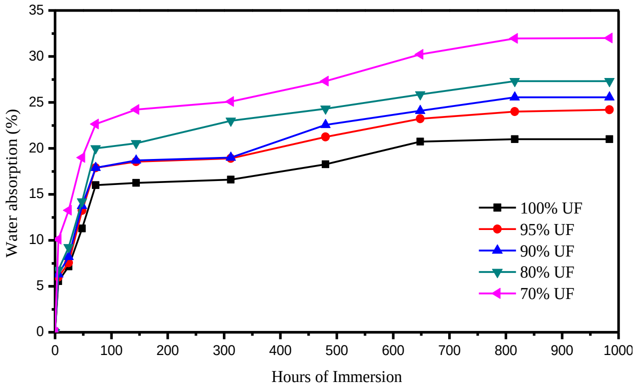


Figure 8. Water absorption of Urea-Formaldehyde/corn husk cellulose

4. Conclusions

Cellulose fibers were successfully extracted from corn husk by chemical modification using mercerization which was confirmed by XRD and FTIR results. Urea-formaldehyde matrix was reinforced with cellulose fiber obtained from corn husk with different composition of composites were prepared *via* solution casting method. The FTIR spectra showed corresponding peak of cellulose and UF resin suggesting well distribution of fiber in polymer. Some shifts in peaks of FTIR suggested feeble chemical interaction between matrix and reinforcement. Prepared composites were analyzed for mechanical property (compressive strength) and it was found that fiber loading increased the mechanical strength of composites upto 30% of fiber loading. Additionally, water absorption test concluded that water uptake of composites increases with increased loading of cellulose which ultimately makes the composite more bio-degradable and eco-friendly.

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