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Development of Antibacterial PVA/CNF Nano-Biocomposite Film Adapting Modified Solution Casting Method

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Abstract:
Solution casting method is the most prominent, effective and cheap method to fabricate thinner nano-biocomposite films at laboratory-scale. But due to improper dispersion of fiber in matrix just by mixing or stirring restricts its applicability. This modified method involves combination of ultrasonication process with solution casting method to fabricate antibacterial PVA/CNF nano-biocomposite film. As a result, agitation of cellulose nano fiber into already prepared polyvinyl alcohol solution increases due to generation of high shear force. A homogeneously dispersed film with enhanced properties is prepared by adapting this technique.

Keywords: *Solution Casting Method, Ultrasonication, Cellulose Nano Fiber (CNF), Nano-Biocomposite Films*

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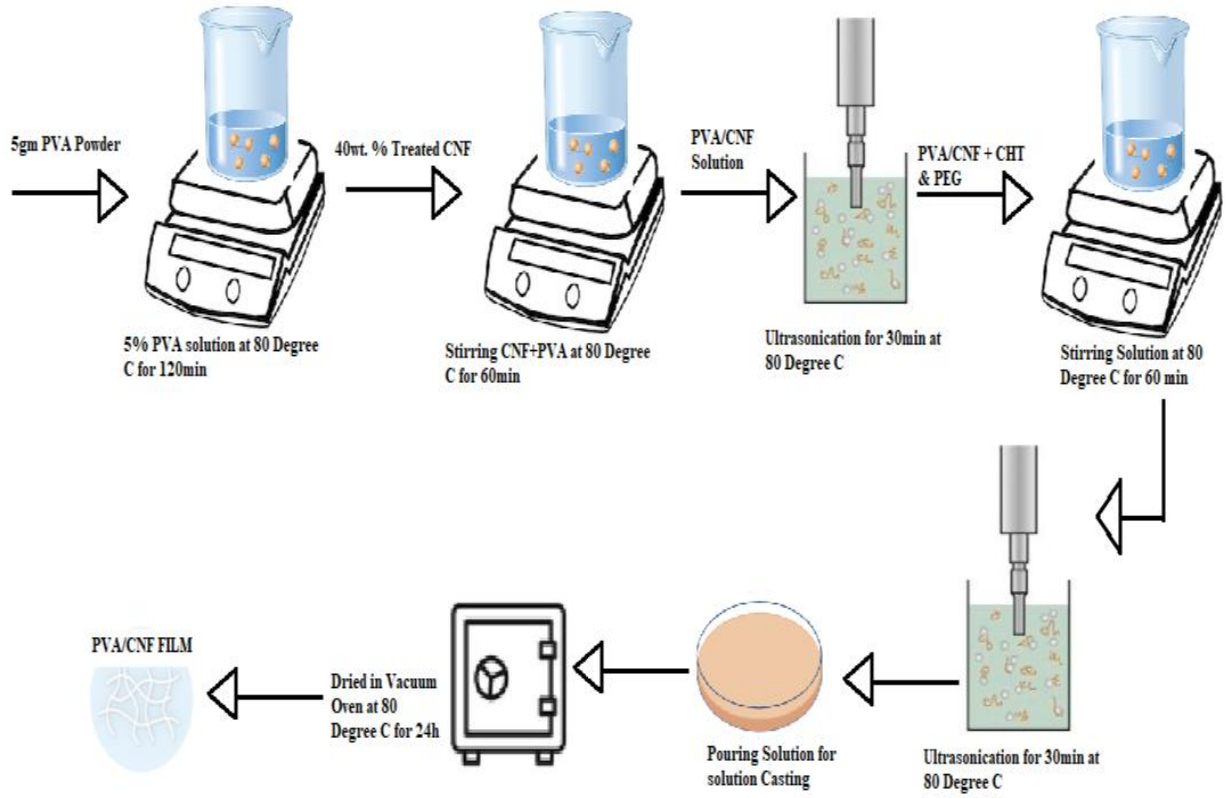
Graphical Abstract:



I. INTRODUCTION:

Awakening of human towards ecosystem sustainability, development of green technologies especially in food packaging industries has become increasingly valuable for mankind. Almost all of the processed food products employed petroleum derived plastics having shallow-life but prolonged in atmosphere for millennia

causing immense threat to environment. In contrast with the past, agricultural residues are appealing source of nano-cellulose which is abundant, ecological and economical replacement of other limited natural as well as synthetic fibers [1]. Cellulose Nano Fibers have outstanding mechanical, thermal and antimicrobial properties, renewability and high applicability after functionalization [2, 3]. Prominent agricultural crop Sugarcane (*Saccharum*) has yield of about 1.8 billion tones per year contains 40-50% cellulose in bagasse which is noticeably much more than various crop residues mainly wheat straw (33wt %), corn cobs (34wt %), corn stalks (35wt %) and rice straw (36wt %) [4-6]. Sugar Cane Bagasse is a low value, plentiful and biodegradable residue that makes it encouraging nano reinforcement in novel green composites [7]. A research study shows that the alkaline and acidic extraction of bagasse from sugar cane yields 56% and 63% cellulose respectively [8]. Solution casting is the primeval fabrication technique invented by Eastman Kodak in the nineteenth century [9]. For the development of nano-biocomposite films solution casting is the simplest yet most flexible method, particularly when used with water-soluble bioplastics. Typically solution casting is a low temperature process offering consistent thickness, less haziness and entropy with high optical clarity [10]. In this process, fiber is incorporated into already prepared biopolymer solution and the biocomposite film is developed by solvent precipitation technique. Steady evaporation often led to accumulation of nano-fibers in the solution resulting non-homogeneous dispersion and poor interaction between them [11]. Even though this approach provides a favorable outcome but with biodegradable hydrophobic polymers the recommendation of this fabrication technique is restricted [12]. The major goal of this research work is to develop nano-biocomposite films utilizing polyvinyl alcohol and cellulose nano fiber from sugarcane bagasse by modifying solution casting method to obtain its suitability in packaging applications [13, 14, 15, 16]. To fabricate nano-biocomposite film slightly different methodology is described and enhanced properties are validated through results.

II. METHODOLOGY

2.1 Materials

Polyvinyl Alcohol Cold (M.W. 850000-124000, 99% degree of hydrolysis and viscosity 23-38 cP) and Polyethylene Glycol 6000 (PEG) (M.M. 5000-7000 with melting range from 56-61^{0}C) were supplied from HPLC, India. Antibacterial agent Chitosan (CHT) (M.W. 3800-20000), Acetic Acid, Sodium Hydroxide were purchased from HIMEDIA, India. Cellulose Nano Fiber (CNF) from Sugarcane Bagasse was transported from Maple Biotech pvt.ltd. India. The materials used are non-toxic, eco-friendly and bio-degradable in nature.

2.2 Functionalization of Cellulose Nano Fibers

Firstly the cellulose nano fiber from sugarcane bagasse was washed in deionized water to remove undesirable particulate matter. It was dried in oven at 50 °C approximately for 8 h and then concealed in packets at room temperature. After that, to eliminate residual lignin, hemicelluloe and other impurities fiber was stirred in hot plate at 250rpm with aqueous solution of acetic acid (5wt. %) at 80^{0}C for 6h. The solution was cleaned with distilled water until 7 pH is obtained, then the solution was filtered and oven-dried at 50^{0}C for 4h. Again aqueous suspension of 5wt. % NaOH was prepared in 500mL distilled water and nano cellulose is refluxed for 24h at 80^{0}C in a magnetic stirrer. The refluxed solid particles were cooled to ambient temperature, and then the CNF were continuously washed until the pH of solution was neutral and centrifuged at 6000rpm for 10min. After 5 washings the centrifugation operation was paused. The functionalized cellulose nano fiber thus obtained was then filtered and oven-dried in vacuum at 50^{0}C for next 24 h and was used as reinforcement in nano-biocomposite film. Functionalization process of sugarcane bagasse CNF is graphically depicted in **Figure 1**.

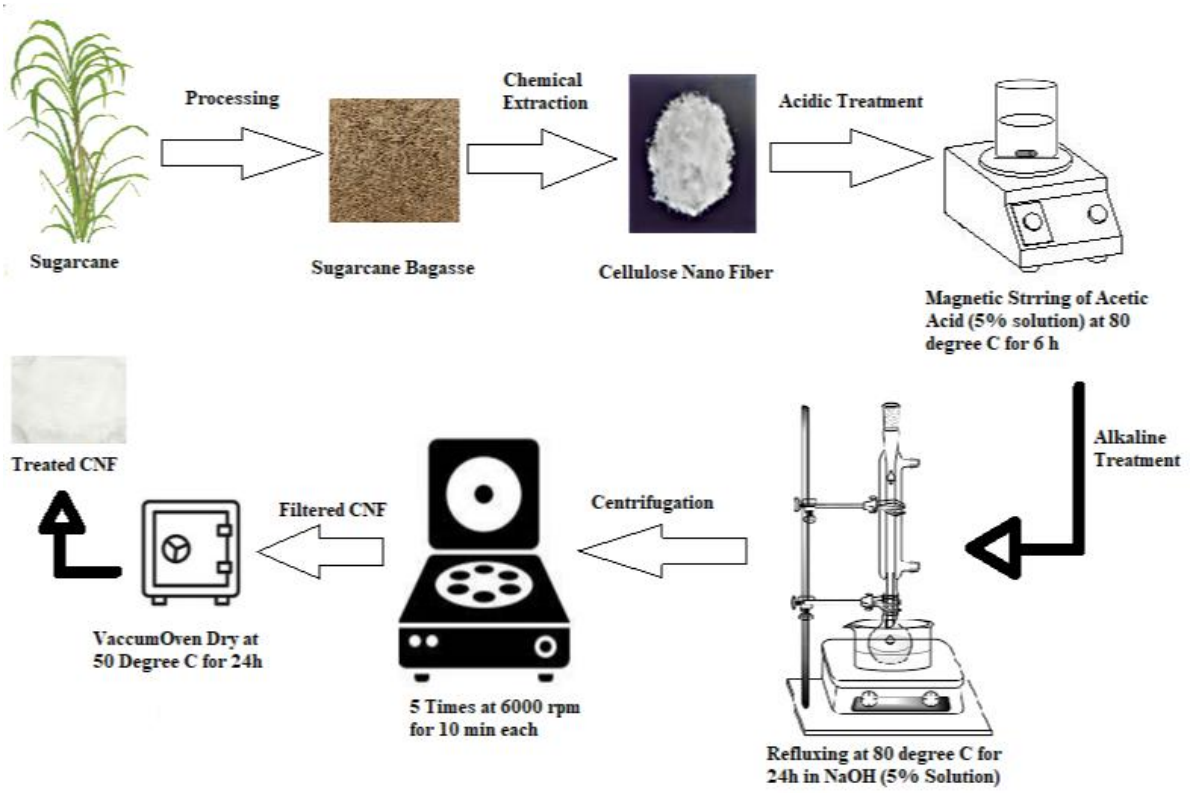


Fig.1 Pictorial Representation of Functionalization of CNF from Sugarcane Bagasse

2.3 Development of Antibacterial PVA/CNF Film

The 5wt. % PVA solution was first prepared by dissolving free-flowing powder of PVA in deionized water at 40^0C by taking 5g of PVA powder in 95mL of water and stirred at 80^0C and 250rpm for 2 h in a hot plate magnetic stirrer. Now the functionalized cellulose nano fibers were added to prepared PVA solution and stirred for another 1h at same condition. Sugarcane bagasse cellulose was added at 30, 40 and 50wt. % respectively to fabricate different nano-biocomposite films. Then obtained solution was sonicated for 30 min at 80^0C and 37% power without time lag in Ultrasonic Homogenizer Probe Sonicator. The 70wt. % Polyethylene Glycol and 1.25gm chiosan were added to mixture and mechanically stirred at same temperature for next 60 min. The final suspensions were sonicated for 30min at same condition to obtained homogeneous solution and remove any entrapped air bubbles. Finally the films were casted in glass petridish and dried in vacuum oven at 80^0C for 24h for complete removal of water. The resulting composite films were then peeled off and kept in the desiccator to equilibrate the films for 24 h before characterization. A detailed flow chart discussing the methodology to fabricate antibacterial PVA/CNF nano-biocomposite film from solution casting method by modifying it with ultrasonication technique is presented in **Figure 2.**

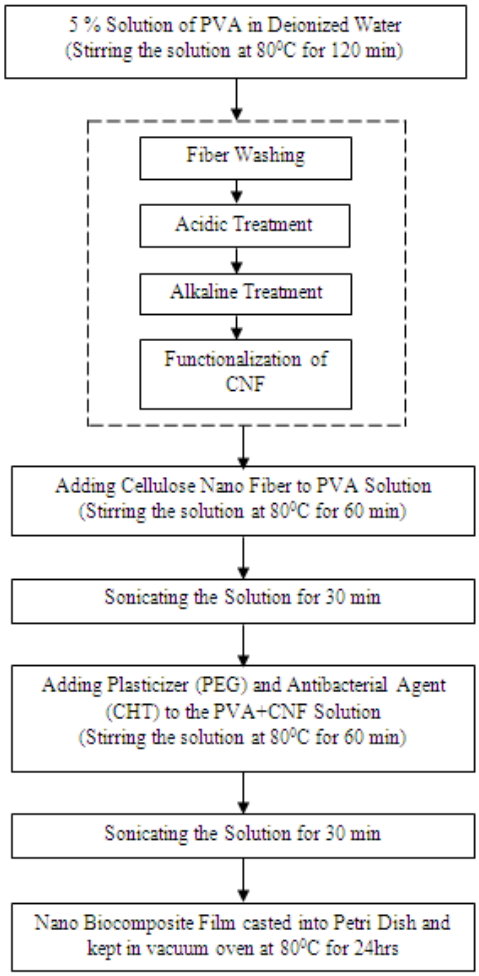


Fig. 2 Development of Antibacterial PVA/CNF Nano-Biocomposite Film Adapting Modified Solution Casting Method

Table 1 represents the nomenclature of fabricated antibacterial nano-biocomposite films from Poly Vinyl Alcohol (PVA) - 5wt. % and treated/untreated Cellulose Nano Fiber (CNF) - 40wt. % blended with Polyethylene Glycol (PEG) - 70wt. % and Chitosan (CHT) - 1.25g by solution casting method as well as adapting modified solution casting method by sonication process.

Table 1 Nomenclature of Developed Nano-Biocomposite Films

Abbreviation	**Full Name**
PVA-1	Pure Poly Vinyl Alcohol Film without Sonication
PVA-2	Pure Poly Vinyl Alcohol Film with Sonication
PVA/CNF-11	PVA & Untreated CNF (40wt. %) Film Blended with PEG and CHT without Sonication
PVA/CNF-21	PVA & Treated CNF (40wt. %) Film Blended with PEG and CHT without Sonication
PVA/CNF-12	PVA & Untreated CNF (40wt. %) Film Blended with PEG and CHT with Sonication
PVA/CNF-22	PVA & Treated CNF (40wt. %) Film Blended with PEG and CHT with Sonication

III. METHOD VALIDATION

Ultimate tensile strength and Elongation at Break (%) of antibacterial PVA & CNF (Treated/Untreated) films reinforced at 40wt. % and blended with PEG, CHT were determine on Universal Testing Machine according to ASTM D882-12 standard. The highest tensile strength was recorded 80.47MPa in treated CNF

followed by 70.13MPa in untreated CNF sample when casted with sonication process. Similarly, highest percentage elongation was found 15.08% followed by 11.97% in the PVA/CNF-22 and PVA/CNF-12 samples respectively. Thus by adapting modified solution casting method the enhancement in mechanical properties was observed irrespective to functionalization of fiber. This may be attributed to good dispersion of fiber in matrix increasing interfacial bonding between them as compared to solution casting method. Moreover, homogeneous films were formed because no cavitations and agglomeration of fiber took place. Mechanical Properties of developed films are presented through Bar Graph in **Figure 3**.

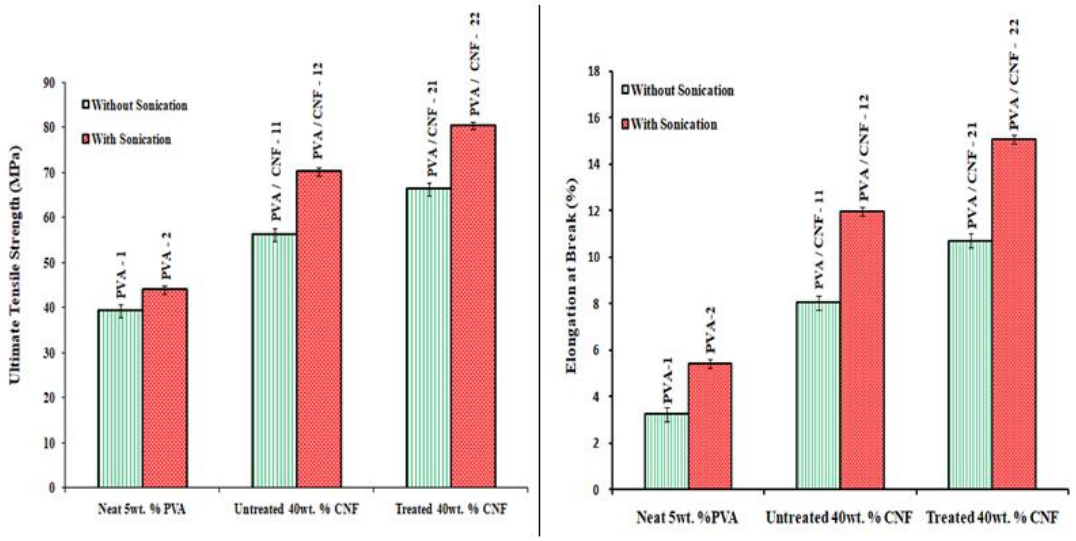


Fig.3 Effect of Sonication on (a) Ultimate Tensile Strength MPa and (b) Elongation at Break (%) of Developed PVA/CNF Films at 40wt. % Reinforcement Treated and Untreated Both

IV. CONCLUSION

Ultrasonication process prevents the re-agglomeration of cellulose nano fiber in polyvinyl alcohol as occurred in solution casting method. Moreover, consistent dispersed solution is prepared without any air bubbles. Thus the developed antibacterial PVA/CNF film by adapting modifying solution casting technique has enhanced mechanical properties. Thus this method can be used effectively in fabrication of nano-biocomposite films.

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