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Development and Characterization of PVA based Green Nanocomposite Membrane for Next Generation Sustainable Food Packaging Applications".

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A biodegradable and non-toxic water-soluble polymer is Polyvinyl Alcohol (PVA) is used in the present work. It is used in production industry applications because of its biocompatibility, low toxicity, and good adhesive properties. Biodegradable composite films are created in the current work using waste materials like wheat husk and coconut fibers. The alkali procedures are used to create the wheat husk (WH) powder. It is low cost and light-weight. Coconut fibers are eco-friendly and non-polluting too. Aloe-vera consists of properties such as adhesive properties, flexural strength, and good tensile strength. The solution-casting approach is used to create composite films with a polyvinyl alcohol basis. Films are given physical and mechanical descriptions. SEM (scanning electron microscopy), FTIR (Fourier Transform Infrared) spectroscopy, and DSC (differential scanning calorimeter) are used to study structural behaviours (DSC). Thermogravimetric analysis is used to evaluate the thermal stability of composite films (TGA). Mechanical properties such as tensile strength, % elongation, young modulus and surface properties have been evaluated. The non-destructive technique of X-Ray diffraction (XRD) is used to characterize crystalline materials. It provides detailed information about a material's crystallographic structure, chemical composition, & physical properties, including as structural parameters such as average grain size, crystallinity, strain, and crystal flaws, as well as crystal orientations (texture). Raman spectroscopy is a non-destructive chemical examination tool that can disclose a material's molecular interactions, chemical structure, phase, and polymorph. It is based on how light interacts with a substance's chemical connections.

Keywords: Polyvinyl Alcohol (PVA), Composites films, Tensile strength-RD, Fourier Transform Infrared (FTIR), Raman spectroscopy

1 Introduction

Polymers with a diverse set of characteristics play an important part in our daily lives[1]. However, as the use of polymeric products grows, so does concern about the environmental impact of these polymers. Traditional polymers are non-biodegradable and are made from fossil feed stocks. In the United States and Western Europe, respectively, around 30 million & 15 million tonnes of plastic waste have been generated[2]. The bulk of conventional plastics & composites are disposed of in landfills at end of their useful lives, where they can remain undisturbed for decades without degrading and rendering them and unusable. Polymers, textiles, and other chemicals account for about 6–7% of all petroleum produced today. We are currently consuming petroleum at “unsustainable” rate, 100,000 times faster than natural world can provide it[3]. In recent years, environmental concerns have compelled creation of environmentally benign, sustainable, & biodegradable polymers & composites[4]. Low density, good mechanical properties, good biocompatibility, & scalability from renewable energy resources are all characteristics of cellulose that have contributed to its growing popularity[5]. Because the cellulose crystal's Young's modulus can reach 134 GPa, cellulose nanocrystals are projected to have a high rigidity[6]. The hydroxyl groups on partially hydrolysed polyvinyl alcohol (PVA) should engage with hydrophilic surfaces of cellulose nanocrystals, resulting in strong hydrogen bonding, which is where processing factors come into play[7]. Nanoscale cellulose fibers and their usage in composite materials have drawn a lot of interest due to their excellent strength and stiffness combined with low weight, biodegradability, and renewability. The use of cellulose nanofibers in polymer reinforcement is a relatively recent subject of investigation[8]. The high rigidity of the cellulose crystal for strengthening is the main driving force behind the use of microcrystalline cellulose in

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composite materials. Tearing complex plant's hierarchical structure into individualised nanofibers with high specific surface area and fewer amorphous sections is one way to do this[9].Cellulose fibers are being employed as prospective reinforcing materials because of their numerous advantages, Including their abundance, low weight, biodegradability, low abrasive character, intriguing particular qualities, & fact that they are waste biomass with good1mechanical properties[10,12]. The drawbacks of cellulose fibers include their poor compatibility with hydrophobic polymer matrixes, moisture absorption, quality fluctuations, and limited heat stability [13,14]. Cellulose is a1polymer, which is a lengthy chain1of smaller molecules bonded together in nature. Sugar D-glucose, forms links in cellulose chain [15]. PVA stands for polyvinyl alcohol and is a semi crystalline polymer. It is water soluble, ethanol soluble, and insoluble in other organic solvents[16]. It consists of good mechanical properties such as tensile strength. Polyvinyl alcohol is tasteless, odourless, and good biodegradable film forming properties[17]. Because of approaching depletion of fossil fuels, new bio-based, ecologically friendly goods are being developed. The utilization of natural filers (biofibers) as load bearing establishes in composite membrane[18]. Increased cellulose content and improved mechanical qualities were obtained from wheat husk, coconut fibres, and aloe-vera. The non-bio-degradable petroleum-based plastics are very harmful for our environment and serious health issues due to non-biodegradability reason behind prepared by bio-degradable and renewable composite film[19]. Their employment in a wide range of applications in the manufacturing sector, medical, pharmaceutical, filtration, & catalysis industries is possible due to their outstanding appropriateness for high mechanical performance and surface functionalization[20]. Wheat, the Yangtze River in China is one of most common crops in the world. 120 million tons of wheat reap annually in China. Wheat residue accounts for 15.7 percent of all post-reap crop residues left in field[21]. Farmers directly burn crop grain in the field since it is simple to access fields that have been prepared for the upcoming crop season. It includes elements that are extremely damaging to the ecosystem, such air pollution. China produces open straw burning estimated 1.036 and PM 2.5 emission per year [22]. PVA is an example of a biodegradable matrix and is commonly used for films made from composites. Polymer is a substance or material made up of very big molecules, or macromolecules, made up of numerous repeating subunits, derived from the Greek words POLY and MER. Poly means "many" & mer means "part". A type of small polymer is a monomer. There are several useful studies on composite based on micro fibrillated or nanosized wood & plant fibers in the previous literature. Nanosized fibers have increased the cellulose content of wheat straw & improved thermal & mechanical properties. Such extraordinary properties render the polymer fibers optimum candidates for many significant applications tissues engineering, encapsulation of cells, Wound dressings, and release of medications[23]. Polyvinyl alcohol (PVA) is a biocompatible, non-toxic synthetic polymer with good chemical, thermal, & hydrophilic properties, along with excellent biocompatibility. It also has a wide range of crystallinity around it. PVA has recently been discovered to be suitable for application in electronics and biotechnology in transition[24].The adverse effects on the environment of our throwaway consumer lifestyle are very evident and also, expressively recorded & as a preventive measure, government is increasingly presenting legislation to normalize harmful environmental effects resulting from supplies used in manufacture of countless common place goods[25]. Several methods for synthesising nanofibers from plant resources are discussed. The integration of macro- and nanofibers as reinforcing materials in the construction of enhanced polymer composites and their application in a number of applications is a possibility. In our work PVA based biodegradable composite films are prepared with natural fibers organic polymer such as wheat husk coconut fibers, aloe-vera with detailed experimental study of XRD, FTIR and mechanical testing investigation. Chemical analysis was used to determine the fibers cellulose, hemicellulose, lignin, and sugar content. To do so, researchers used Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and thermogravimetric analysis (TGA). The main objective to make a polymer nano composite membrane out of recycled natural nano composites made from waste material. It is biodegradable. Nanocomposite is a solid multiphase material with nanoscale repeat intervals b/w various phases that make up material in one of stages in 1, 2, or 3 dimensions of less than 100 nanometres (nm). Nanocomposite is a concept that aims to develop and manufacture new materials with unrivalled endurance and physical qualities utilising nanometre-scale building blocks. Nanocomposites-based polymers are compositions in which nanoparticle thermoplastic polymers spread in at least one or two dimension in an organic polymer matrix to enhance better attributes. A nanocomposites film is made up of a fiber and a binder substance (matrix). This combination combines fiber and matrix qualities to generate a new material that may be stronger than the fiber alone. Heterogeneous/hybrid materials known as nanocomposites are produced at the nanometric scale by fusing polymers with inorganic particles. Their architectures have been found to be more intricate than those of micro composites.

2 Materials and Methods

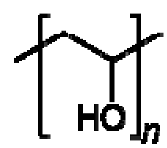
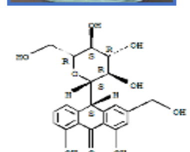
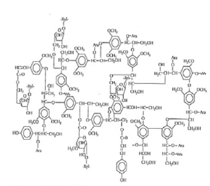
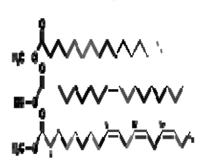
Polyvinyl alcohol polymer (PVA) is a polyethene polymer which is also known as polyvinyl Alcohol. PVA (polyvinyl alcohol) is a synthetic polymer that is water soluble. Polyvinyl alcohol is a material that forms films, polymerizes, and attracts to other molecules. Polyvinyl alcohol (PVA) is a transparent synthetic resin that is water-soluble and is commonly used in the textile and paper treatment industries. Aloe-vera is a type of plant that is used to treat a variety of ailments. Aloe vera is an African native plant. The water content of this plant is 96 percent. This plant's leaf includes more than 75 nutrients and 200 active substances, including minerals amino acids, and vitamins. Details of material used in the present work is given in Table 1.

Mechanical parameters like as adhesion, flexural strength, and longitudinal tensile strength are also included. In the circumferential direction of the cell, the protective barrier, buffer part, and translucent inner layer are visible. In 2015, Wheat husk was harvested in Shandong's Huantai experimental fields. China’s province cellulose, hemicelluloses, lignin, and water make up the chemical composition. Cellulose, hemi-cellulose, and lignin, the three main polymer components, make up 44.81 0.67 %, 34.11 0.75 %, and 8.75 0.31 % of wheat husk, respectively. Wheat husk dry stalks are an agricultural by product collected after the grains have been removed. It has a compact structure with 35-40% cellulose and 30-38% hemicellulose. It contains Mechanical properties, low cost, and light weight. Before being used, the straw was dried & processed into a powder with a particle size of less than one mm. In the studies, only distilled water was used. Coconut fiber is a natural fiber obtained from the outer layer of the coconut husk. Coir is a fibrous fiber found b/w hard & outer coats of a coconut. Brown coir can also be used for sacking and horticulture (produced1from ripe coconut). Unripe coconuts are used to make white coir, which is used to make finer brushes, thread, rope, and fishing nets. It is stable and does not sink, allowing it to be used for vast distances in deep water without pulling boats or buoys below.

2.1 Preparations of Nanocomposite Membranes

The solution cast process was used to create composite films on a basis of polyvinyl alcohol (PVA). The powder form of 1 gm PVA and 10 ml distilled water dissolved in conical flask. Homogenous solution by stirring at magnetic starrier machine. The process started at room temperature 20°C to 22°C then slowly increased the temperature at 80°C. The solution will be stirrer at fully transparent then temperature slowly reduced at 80°C to room temperature 20°C to 22°C For 1 hour. Thereafter the solution was poured into a glass petri dish. After drying solution at room temperature for seven days, the films were removed from the petri dishes. Polyvinyl alcohol and aloe-vera composite film is prepared from previous method. 5 gm aloe-vera gel is stirred at 1 hour and stirrer at polyvinyl alcohol powder with distilled water and both solutions are mixed. The solution was again stirred for 1 hour and completely transparent homogenous solution. There after the solution was poured into petri dish at room

Table 1 — Detail of material used in Nanocomposite Membranes

Material	***Chemical formula***	
PVA (Poly Vinyl alcohol)		
Aloe vera		
Wheat Husk		
Coconut fiber		

temperature 20°C to 22°C. After drying the solution at room temperature for seven days, the films were removed from the petri dish. Three to four washings in HCL solution were performed on coconut fibers. Using a magnetic stirrer machine for 30 minutes with 400 ml of distilled water and 32 pallets of sodium hydroxide NaOH solution stirrer. A prepared NAOH solution was dipped into the coconut fibers for one hour. Coconut fibers were cleaned three to four times before being placed in an electric conventional oven set at 80°C for 15 minutes. The PVA and aloe vera solution was made using the solution cast method and then poured into coconut fibers and a petri dish. After being dried for 7 days the composites films were removed from the petri dish. Wheat husk small particle were separated from constitutes longer wheat fiber and dipped into water at 15 minutes then washed two times for impurities and dust particles were removed. Once more, immerse wheat fiber was immersed for two hours in HCL solution. Wheat fibers were washed several times then put the electrical conventional oven 80+5^{0}C.The sodium hydroxide solution with distilled water 400ml distilled water and 32 palettes NaOH tablets were stirred at magnetic stirrer machine. Then wheat fibers were dipped into the NaOH solution for 4 hours. Then wheat fibers were washed 3to 4 times and was put into the conventional oven. Pore size 100 ASTM sieves are used to separate the wheat fiber from wheat husk powder. Finally, wheat powder was prepared by alkali method. 50% PVA + aloe-vera and 50% wheat husk powder stirring at the magnetic stirrer machine at the room temperature for 2 hours. The solution was poured into petri dish for 7 days. The composites films were removed from the petri dish. Various preparation methods of PVA nanocomposites is shown in Fig. 1 and prepared samples are shown in Fig. 2.

3 Results and Discussion

Different nanocomposite membranes were prepared in this section with polyvinyl alcohol reinforced with nanoparticles of Aloe-vera gel, coconut fibers and wheat husk. Mechanical properties are assessed, such as tensile power, percent elongation and young modulus. The mechanical properties of a natural fiber nanocomposite membrane depend upon orientation and combination of polymer used PVA and natural fibers of fillers such as aloe vera gel, coconut fibers, and wheat husk powder. The stress strain diagram shown in below for various tensile and elastic properties such as Figure 3 (a) Shows the maximum tensile strength for pure PVA nanocomposites membrane was 5.56 MPA. It was

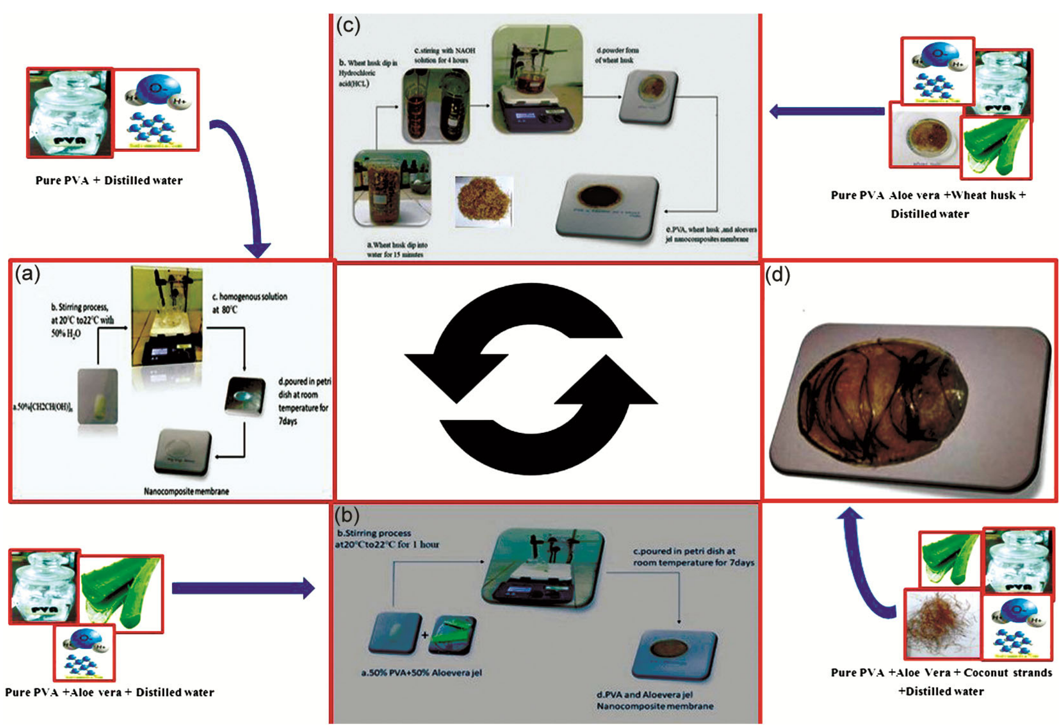


Fig. 1 — Various preparation methods of PVA based nanocomposites for Food Packaging and Schematic illustration of the fabrication process of the different composite film.

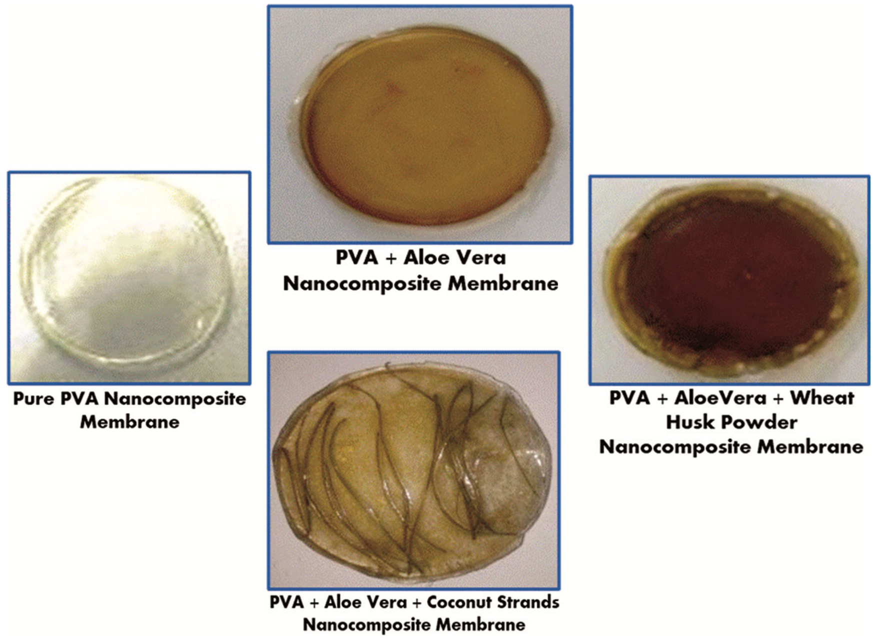


Fig. 2 — Four new class based synthesized natural nanocomposites systems.

observed that membrane elastic region at 0 to 65 % then it was observed that strain to fracture membrane was 0 to 73. Figure3, (b) shows the maximum tensile strength for pure PVA and aloe-vera gel membrane was 10 MPa. In this region, the membrane shows elastic behaviour in nature of the composite's membrane & plastic region shows the 230 % to 305 %, and then fracture the membrane was 11 MPa. Figure 3, (c) shows that, the stress stain graph for pure PVA, aloe -vera gel and coconut fiber better result represent such as good tensile strength, elastic behaviour, plastic region and fracture point. The maximum tensile strength was 6.6 MPa. An elastic region shows that 0 to 7.5 percentages and plastic region shows that 0 to 32 percentages.

Figure 3 (d) shows that normal behaviour represents the PVA, aloe-vera gel and Wheat husk nano powder nanocomposites membrane shows the stress strain relationship at elastic regions of 0 to 23 percentages and plastic region of composite membrane 23 to 25 percentage, maximum tensile strength of the wheat husk nanocomposites 14 MPa. As a result, the wheat husk membrane has the best performance and the highest tensile strength when compared to the membranes made of coconut fiber, jute fiber, and pure polyvinyl alcohol.

The X-ray diffraction patterns of various composite membranes are shown in Fig. 4 below. The composite membrane graph shows (a) black color of pure PVA composites membrane in pure PVA five intense characteristic peaks located at 19.19°, 22.75°, 39.26°,75.6° and 80.66°. Crystalline property shows peak at 19.19°pure PVA decreasing the peak. Then Amorphous behavior showed peak at 20° to 22. 75°. Again shows the crystalline nature 40.75°. The pure PVA film showed the amorphous behavior showed the peak at 80°. In graph 4(b) red color polyvinyl alcohol and jute fiber nanocomposite membrane three intense peak at 19°, 20.88° and 48.06°.

The nanocomposite membrane shows the behavior of crystalline nature peak at 19° and amorphous property showed that peat at 21° to 40. 53°. In blue color shows graph 4 (c) that PVA and aloe-vera nanocomposite membrane properties shown. There is various peak shown such as 19.81°,23.9°,30°,32° 36°,40°,44°,48°,49°, 58° and 61°. Crystalline nature show peak at 19.81° and show amorphous behavior show in figure peak at 44° to 61°and crystalline nature Shows that 36 °to 44°. In graph 4(d) pink color showed that PVA, aloe-vera gel and wheat husk powder membrane. There are three intense peak 19.25°, 20°and 23°. The crystalline property has shown that peak at 19° and amorphous property peak at 23° to 65°.

There are four nanocomposite membrane crystalline and amorphous studies by the XRD data in comparison between all of membrane. The pure PVA nanocomposite membrane highly crystalline behaviors shows the diagram then they also shown PVA and aloe vera gel nanocomposite membrane

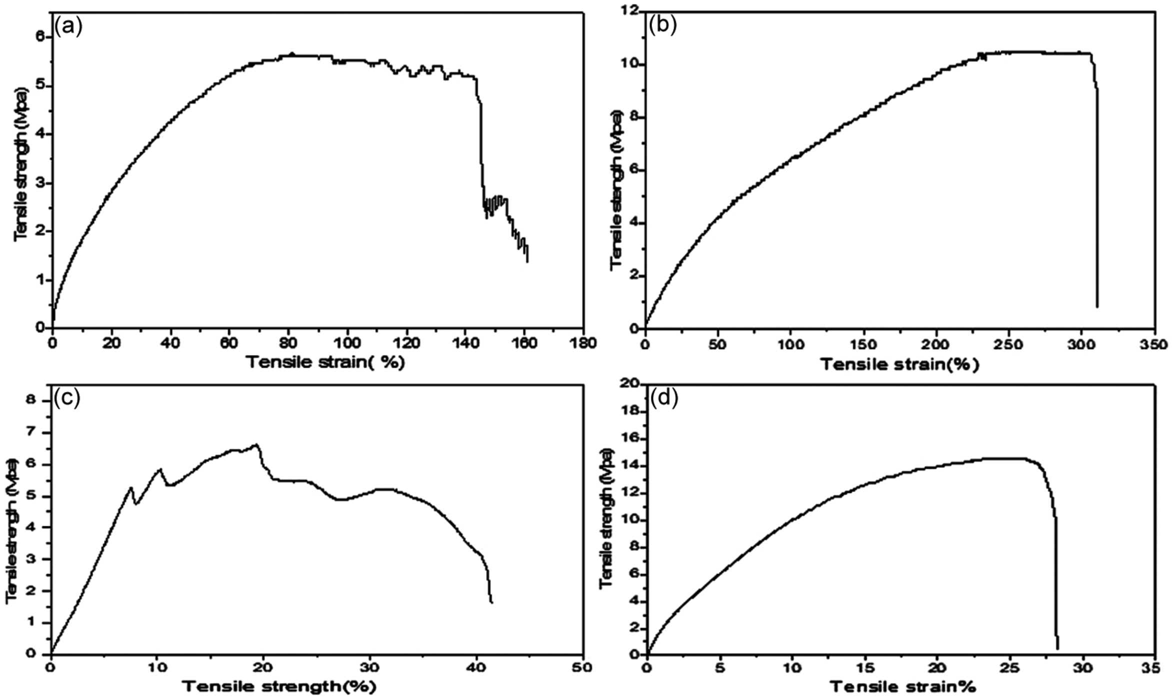


Fig. 3 — Tensile Strength of different nanocomposite membrane (a) pure PVA (b) Pure PVA, Aloe vera gel (c) Pure PVA, Aloe vera gel and coconut fiber (d) Pure PVA, aloe vera gel and wheat husk powder.

crystalline nature and showed that same property of the amorphous behaviors peak at 60° to 80°. In our study Pure PVA does not proper crystalline nature but the PVA and wheat husk nanocomposite membrane showed that as maximum crystalline nature shows as compare to pure PVA, PVA and jute fiber and represent the good amorphous behaviors wheat husk powder nanocomposites membrane. The IR spectrum of NCPE shows formation of Composite films are pure PVA, Coconut fiber, Aloe vera gel, and Wheat husk membranes are shown in Fig. 5. The composite membrane graph shows black color of Fig. 5(a) pure PVA composites membrane. The main feature of Polyvinyl alcohol is appearance of OH hydroxyl vibrational band at 3298.44 cm^{-1} These bands in PVA gets displaced towards at the lower wave number and OH stretching vibrational peak at 2910.85 cm^{-1}.

The hydroxyl bands are very complex. OH, stretching behaviors of peak in the range of 2844.96 - 1574 cm-1. In Fig. 5(b) Both PVA and Coconut fiber membrane showed an asymmetric CH_2 twisting and vibrational frequency increase peak in range of 1031-833 cm^{-1}. Strong peak corresponds to 1327.51 cm^{-1} is associated with C-H and plane bending, C=C stretch, wherein from the theoretical aspect, the O-H bending vibration becomes weak gradually from mono to tetra- molecular configuration. Blue color in Fig. 5(c) the IR spectrum of NCPE show formation of both composites of PVA and aloe vera membrane shown in Figure. The peak at 3289.72 cm^{-1} of PVA and aloe-vera film O-H stretching vibration. The crystalline tendency increases at the peak of 2844.96-2927.32 cm^{-1}. The peak at 1722.868cm^{-1} will be stretching tendency and C=O. The change of vibrational properties such as bending zone in the range of1566.86 -833.33cm^{-1}. The strong peak at 1426.35 cm^{-1}represents with CH_2 plane scissoring C-H and O-H in -plane bending C=C asymmetric stretching vibration. There also exits a weak peak correspond to 1566.86cm^{-1}. In Fig. 5(d) The PVA, aloe vera gel, and wheat husk powder composite film showed peaks 3287.23,2910.46, 1726.06,1643.61, 1560.28,1416.66, 1379.43,1265.95, 1100,911.34 cm^{-1}. Composite film peak at 3287.23 cm^{-1} broad relates to 1C-H stretching and CH_2 asymmetric stretching vibration and O-H stretching behaviour of peak at 2910.46cm^{-1}. The peak at 1726.46cm^{-1} will be stretching tendency. The small peak at 1100cm^{-1} associated with C-O stretching, CH_2 in plane rocking, and O-H in plane bending.

The Raman spectra of four different composite films are shown in figure below-6(a), (b), (c) and (d). The diagram represented structural behaviour of membrane at room temperature. The comparative

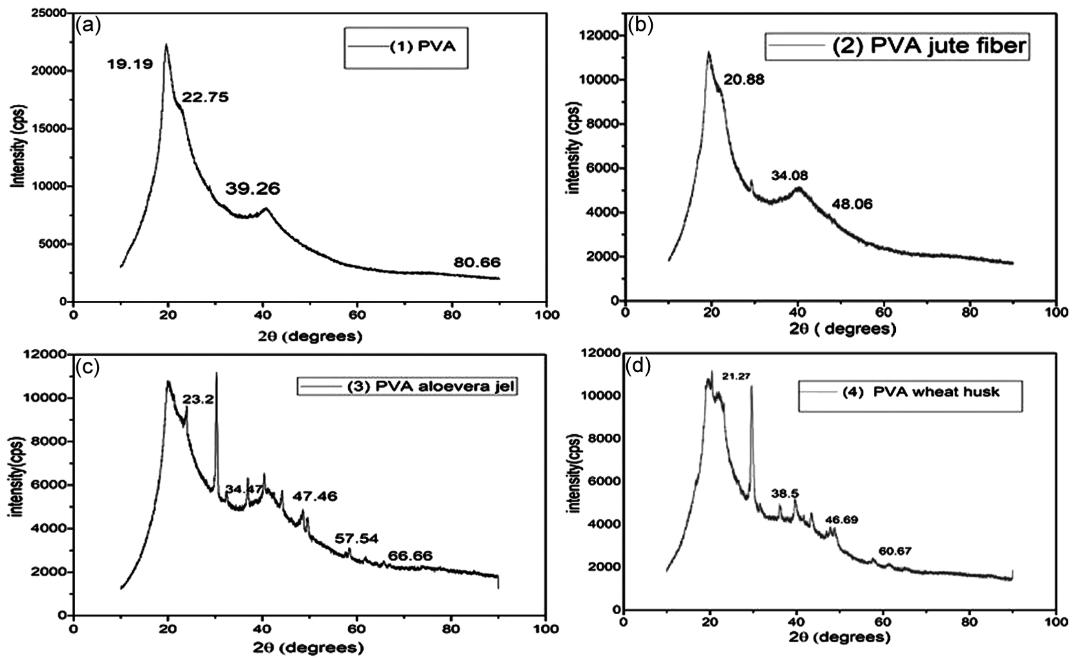


Fig. 4 — X-ray diffraction study of different nanocomposite membrane (a) pure PVA, (b) Pure PVA, Aloe vera gel and coconut/jute fiber, (c) Pure PVA, Aloe vera gel, and (d) Pure PVA, aloe vera gel and wheat husk powder.

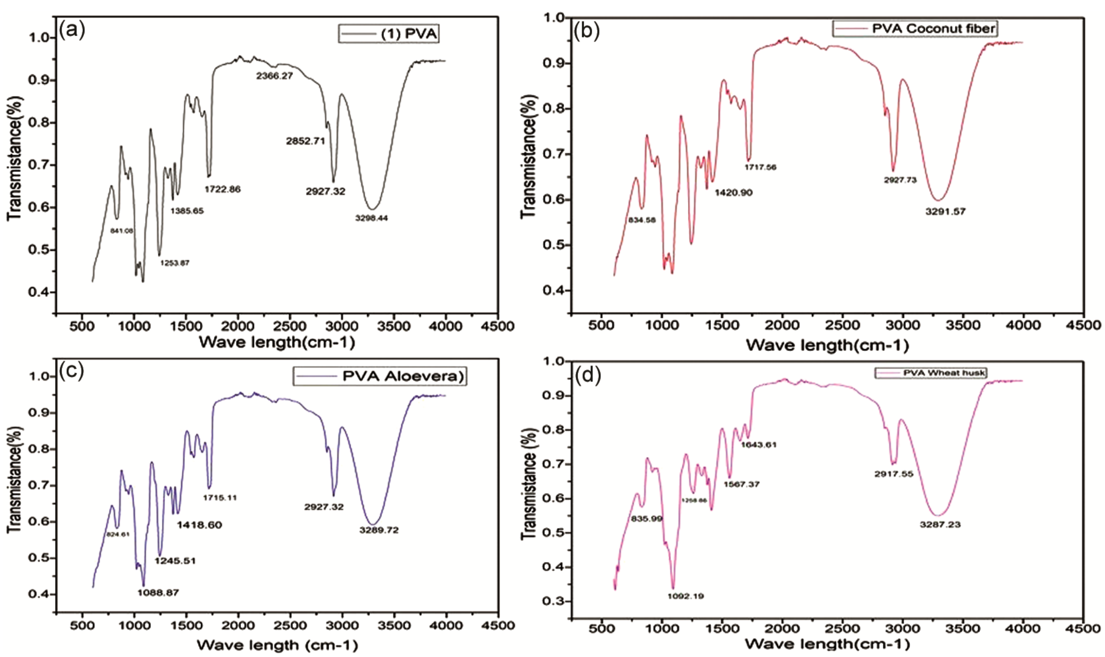


Fig. 5 — FTIR spectroscopic study of different nanocomposite membrane (a) pure PVA, (b) Pure PVA, Aloe vera gel and coconut/jute fiber, (c) Pure PVA, Aloe vera gel, and (d) Pure PVA, aloe vera gel and wheat husk powder.

studies of the vibrational peak obtained from both theoretical models and experimental results are described in Fig. 7. Figure 6(e) show that the black color pure PVA nanocomposite film is vibrational studies here. From the observation, at room temperature, the experimental peak at 1024.45cm^{-1} and 1074.50cm^{-1} is a bulk structure is formed due to cross linking of polymer chain. The bulk structure shows that high isotropic conductivity, and its polarizability is virtually dependent on poorly bound electrons. The peak at 1504.61cm^{-1} also represented the vibration of PVA closer to which disappears at higher temperature.

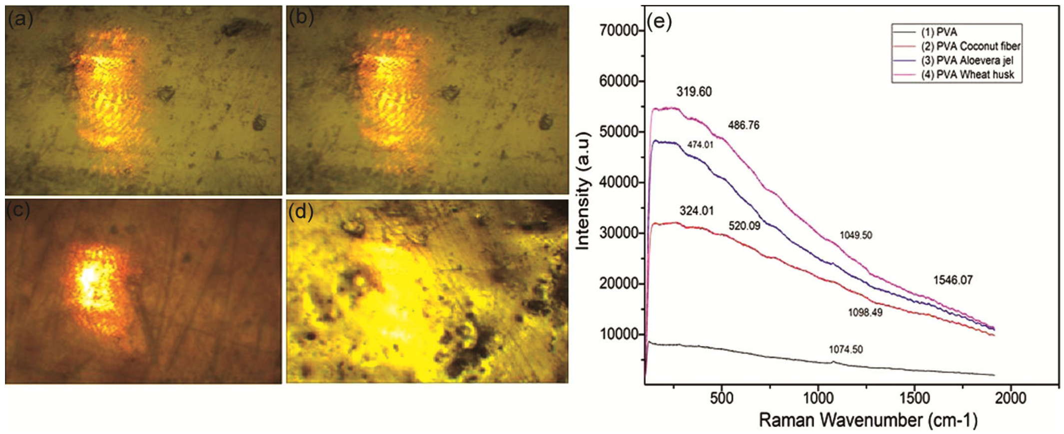


Fig. 6 — RAMAN spectroscopic study of different nanocomposite membrane (a) pure PVA (b) Pure PVA, Aloe vera gel and coconut/jute fiber, (c) Pure PVA, Aloe vera gel, and (d) Pure PVA, aloe vera gel and wheat husk powder (e) Raman spectral analysis.

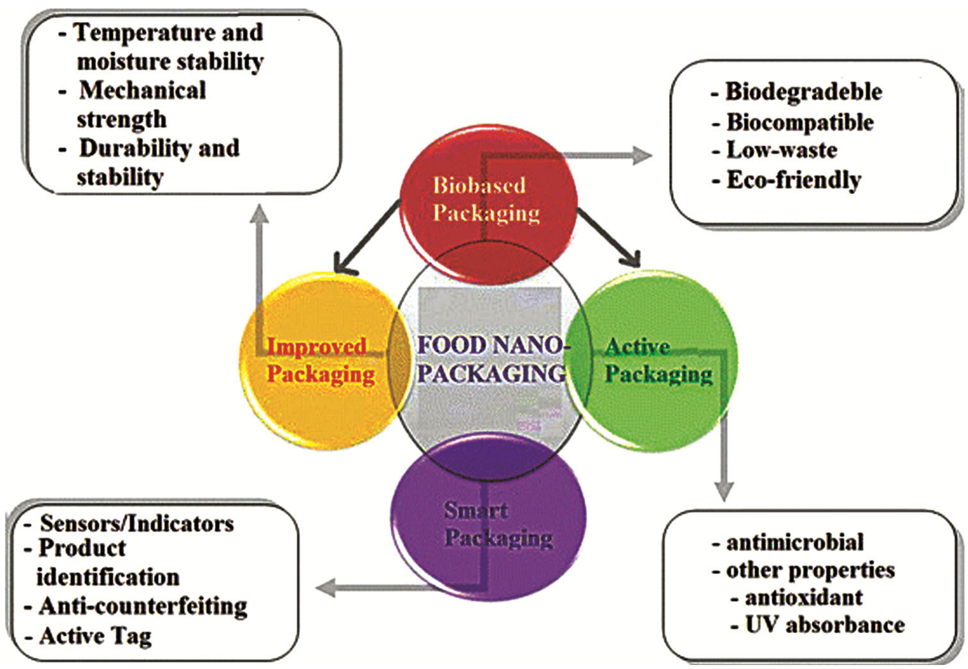


Fig. 7 — Detail Forms of Food packaging in different fields.

Figure 6(c) shows the red color represent of composite film is PVA and coconut fiber membrane peak located at 1098.49cm^{-1}.

It represents C-O stretch, CH_2 in plane rocking O-H in plane bending. The blue color composite membrane our observation peat at 478.62cm^{-1}arise O-H interaction between Pure PVA and aloe-vera gel molecules. At room temperature another two peaks are found at 177.49cm^{-1}. The pink color also shows that composite membrane of PVA, aloe vera and wheat husk powder. In our observation of peak at 1049.50,1278.92, 1546.1cm^{-1}. Our observation the clear vibrational molecular structure showed that wheat husk nanocomposites membrane.

3.1 Application of Biodegradable Composite Membrane:

PVA can also be used as a packaging agent for food supplements and poses no health risks as it is non-toxic. One of leading industrial uses of PVA is for food packaging, which accounted for 31.4percent of global share in 2016. PVA is FDA approved and flexible for food packaging as well as a GRAS (generally recognized as safe) polymer. Packaging requires a very thin thickness of PVA and composite.

The growth and multiplication of bacteria are main reason for food spoilage & presence and absence of oxygen & level of concentration of bacteria are the necessary conditions for survival and reproduction. This necessitates the use of packing materials with

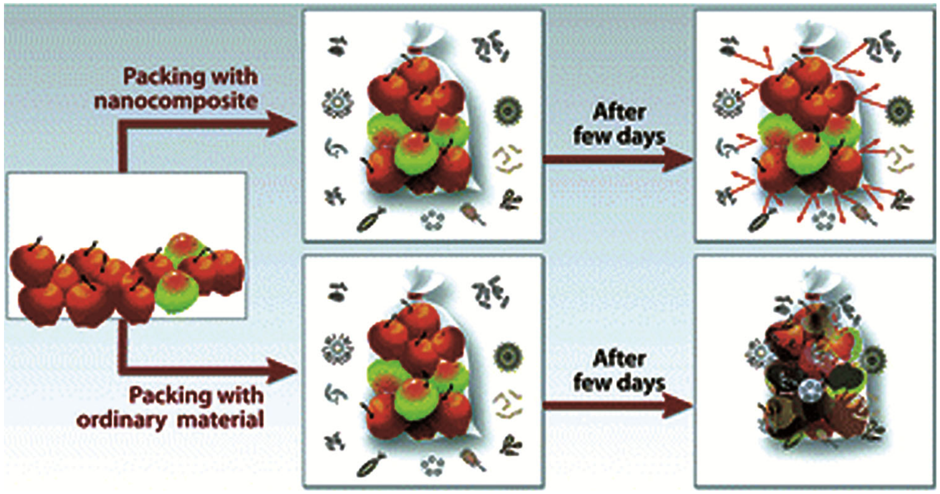


Fig. 8 — Importance of Biodegradable packaging in respect of bacterial growth.

high oxygen barrier properties. Many foods must be stored in a dry state, with no moisture escaping the packaging, and many foods must be controlled in water within a specified range to retain their natural flavour. As a result, the food packing material must have an excellent water vapour barrier performance. To prevent the odour fleeing into the food and also prevent the volatile flavour of food itself.

So, the packaging material must have excellent taste barrier capability. Some items must also be UV light resistant; for example, oil in light will create acids, resulting in product deterioration and flavour degradation. The color of prints & goods can also be affected by sunlight. In addition, sunlight can promote oxidation of fats, alkaloids drugs, Vit B1, B2, Vit C, etc., but also because they quickly respond to the role of light and oxygen, there is a change in color and the reduction of other changes. Here, therefore, they require packaging materials that can be manufactured with a UV blocking function.

4 Conclusion

Biological and biomechanical behaviour of composite films based on PVA filler can be seen using several experimental methods such as SEM, XRD, and IR.XRD observations conform the formation of 12° to 43° amorphous behaviour studied and crystalline size varying between 44 to 45. IR detailed studied with PVA film is vibrational behaviour such as stretching, bending and skeleton features. Different experimental tools, i.e., Mechanical testing, XRD, FTIR and Raman spectroscopy. The mechanical properties of a natural fiber nanocomposite membrane shows that Elastic and plastic behaviour of polymer composites. As a consequence, four nano composites membranes have better outcomes and have the highest tensile strength when compared to coconut fibre, jute fibre, and pure polyvinyl alcohol alone. These membranes are made of PVA, aloe-vera gel, and wheat husk. Pure Polyvinyl Alcohol (PVA), Aloe Vera Gel, and Jute Fiber Nanocomposite Membrane have good tensile characteristics. Crystallographic structure, chemical composition, and physical properties of materials such as crystal orientations (texture), structural parameter1average grain size, crystallinity, strain, & crystal defects are all precise information. The X-diffraction pattern of pure PVA liquid shown in figure 4. Results of the composite membrane graph shows in black color of pure PVA composites membrane. In pure PVA three intense characteristic peaks located at 19°and along with large peak at 20. 29° amorphous behavior showed peak at 23° to 32° and the composite membrane attained maximum crystalline property at 40.53°C and further noticed that at 43 to 90° crystalline property behavior. XRD and FTIR show physical and morphological behaviour of composite films based on PVA filler. XRD observations confirm the formation of 12° to 43° amorphous behaviour studied and crystalline size varying between 44° to 45°. IR detailed studied with PVA film is vibrational behaviour such as stretching, bending and skeleton features. The polymer composites are better use such as Food packaging, medical packaging cover and Industrial packaging cover.

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