“PREPARATION AND CHARACTERIZATION OF PVA CROSSLINKED FILM WITH CINNAMALDEHYDE”

A Thesis submitted in the partial fulfilment for Degree of Master of Technology

By

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CANDIDATE'S DECLARATION

I hereby, declare that the work, which is being presented in this end term dissertation report, entitled "**PREPARATION, CHARACTERIZTION OF POLYVINYL ALCOHOL CROSSLINKED WITH CINNAMALDEHYDE FILM** " was carried out by me during the period June 2016-May 2016 as a part of the partial fulfilment of my M. Tech dissertation, under the supervision of **Dr. Ashish A. Kadam** assistant Professor, Department of Paper Technology, Indian Institute of Technology Roorkee The matter embodied in this dissertation report has not been submitted by me for the award of any other degree in any other institute.

Dated Amrit Singh

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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I am deeply indebted to my supervisor **Dr. Ashish A. Kadam** (Assistant Professor Department of Paper Technology, IIT Roorkee) for inspiring me to enhance my knowledge by pursuing the innovative area of my dissertation work "**preparation, characterization of polyvinyl alcohol crosslinked with cinnamaldehyde film**". Their skill, full guidance and deep knowledge of the subject made the interpretation of seemingly difficult work look easy.

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ABSTRACT

This thesis report is based on the research in the field of crosslinking of polyvinylalcohol with cinnamaldehyde and their application in food grade packaging polymer. It was found that crosslinking of PVA with CIN has more benefit then uncrosslinked film formation. Crosslinked film leads the enhancement of properties such as water barrier , antimicrobial , optical , and mechanical properties raise in dispersible property, gain thermal and chemical stability.

In the present experiment preparation and characterisation of pva crosslinked with cinnamaldehyde film using HCl studied. Prepared PVA crosslinked , uncrosslinked, pure PVA film were characterised by SEM, FTIR, Color test, Water absorbance ,WVTR. Further properties are investigated by polymer matrix using FTIR, and DSC-TGA.

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NOMENCLATURE

HCL	-	Hydrochloric acid
CIN	-	Cinnamaldehyde
PVA	-	Polyvinyl alcohol
SEM	-	Scanning Electron Microscopy
XRD	-	X-Ray Diffraction
FTIR	-	Fourier Transform Infrared Spectroscopy
ASTM	-	American Society for testing and material
TGA	-	Thermo Gravimetric Analysis
WVTR	-	Water vapour transmission rate
DSC	-	Differential scanning calorimetry
Tg	-	Glass temperature

Chapter- 1

1.Introduction

This is the era of development and research of biodegradable food packaging product. Nowadays every product comes in packaging either beverages, horticulture, medicines meat, product. Food packaging is big issue for companies because they spoil in short time due to moisture and gases present in environment. Polymers have good barrier properties against gases and moisture but most of the polymers are non biodegradable so all companies want replacement of non biodegradable to biodegradable polymer.

1.1 Polyvinyl alcohol

It also called as PVOH and PVA is a synthetic polymer soluble in water it has good property to form film. PVA has no odour, non toxic and resistant to grease, oil and solvents PVA has good oxygen barrier property but low moisture barrier. Structure and physical view of polyvinyl alcohol

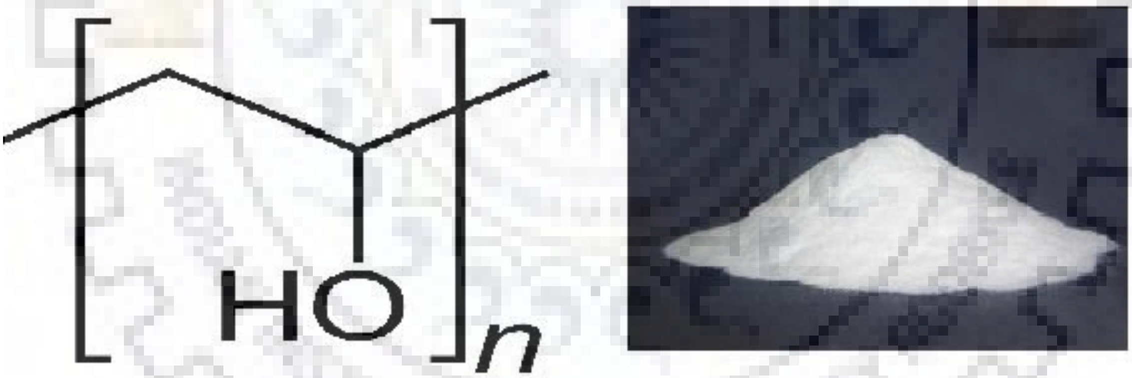


Fig: 1 (a) PVA chemical formula (b) PVA

PVA produced by partial or complete hydrolysis of polyvinyl acetate groups which consist of partial replacement of the ester group in vinyl acetate with the hydroxyl group, completed in the presence of the aqueous sodium hydroxide.

Uses of PVA

Polyvinyl alcohol mostly used in textile and yarn industry for resistant to oil and grease. It also used in fishing sport due to its water soluble nature PVA filled in small bags hanging on hook so fishes are attract and easily catched by fisherman .

Polyvinyl alcohol also used in pharmaceutical industry to coating medicines and used as a coating agent for food supplement.

1.2 Cinnamaldehyde

Cinnamaldehyde is an organic compound of aldehyde group having pungent flavour and odor it makes from cinnamon trees. It is natural compound has yellow color and oily in nature.

Molecular weight of cinnamaldehyde is - 132

Chemical structure of cinnamaldehyde - C_9H_8O

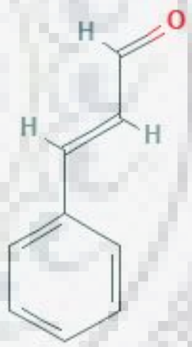


Fig:2 (a)cinnamadehyde (b)chemical formula of CIN

Cinnamaldehyde is also used as a antifungus. To a lesser extent, CIN is an effective insecticide, and its scent is also known to repel animals like cats and dogs. CIN has best antimicrobial properties against both Gram-Positive and Gram-Negative bacteria as well as best effective against fungi and mold.

1.3 Deionised water

Deionised water is a pure form of water does not contain any type of ions accept H^+ OR OH^- ion. It is similar to distilled water but both have different strategy to produce distilled water produce by boiling ,evaporation and condensation of water on the other hand deionised water produce reverse osmosis, ions separation method these ions separate by applying H^+ ions for anion separation (chloride, sulphate, nitrate etc) and OH^- ion for cation separation (manganese, iron, calcium sodium) we get only pure water including only two ions OH^- and H^+

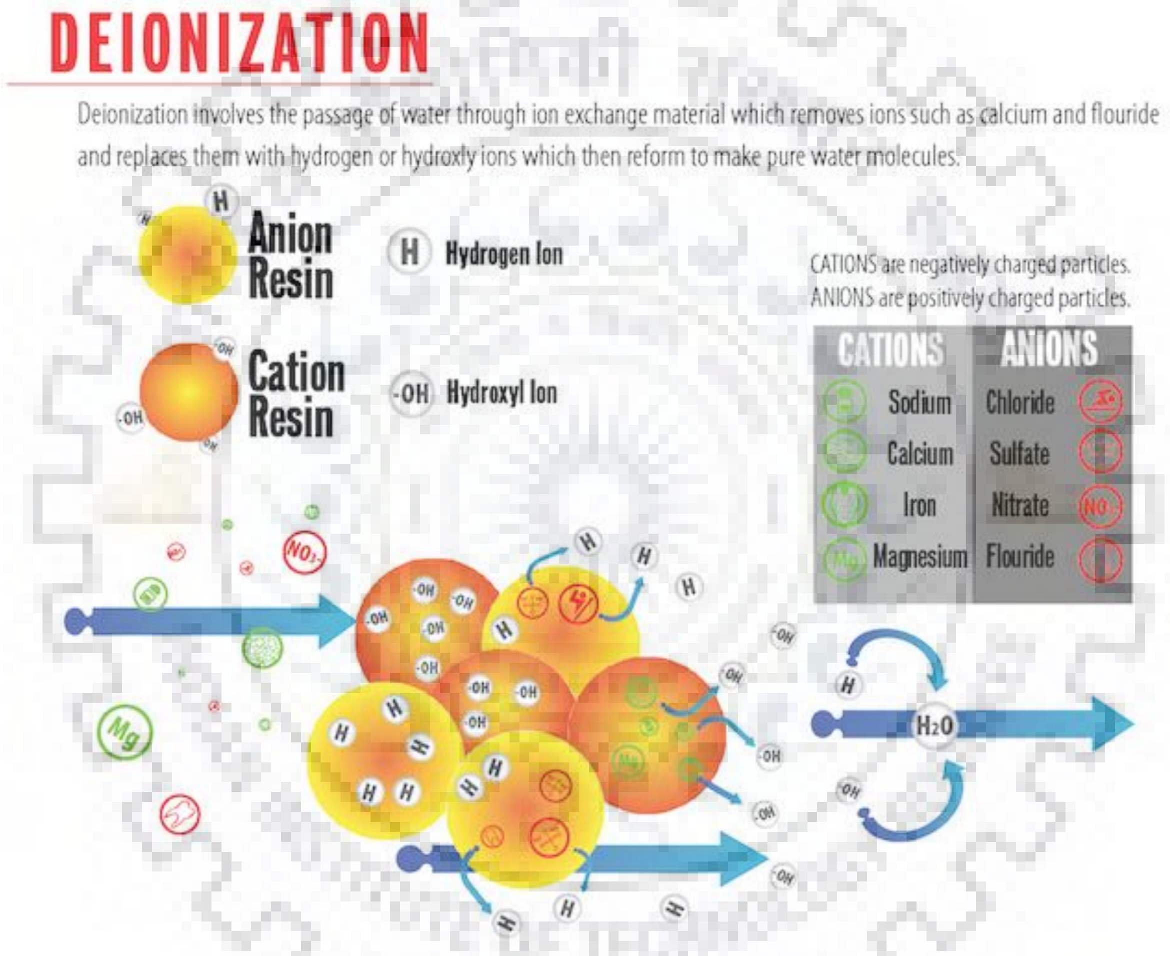


Fig:3. Deionised water ions process

1.4 Hydrochloric acid

It is a strong acid pungent smell colourless look like water used for the maintain pH of solution. HCl is a solution of chlorine and water and inorganic in nature. in industries it used as a chemical reagent. Some uses of HCl

- In domestic purpose diluted HCl used for toilet cleaning and for deskilling agent.
- In food industry HCl used for production of gelatin and food additives.
- HCl also used in leather production industries.

1.5 What is crosslinking?

it is common method to used change physical properties of chemical compound. In this report we are using chemical reaction between polyvinyl alcohol and cinnamaldehyde because PVA is a hydrophilic compound polymer so easily absorb water and dissolve rapidly due to hydroxyl groups present in its tail. To remove hydroxyl group from its tail we are using crosslink reaction with CIN. CIN is a hydrophobic compound less soluble in water.

1.6.1 Advantages of crosslinking

- We can improve mechanical properties of polymers.
- We can change hydrophilic properties of PVA by using some oils.
- We can improve Antimicrobial properties of PVA.
- We can improve thermal properties of PVA.
- We can also improve gas and moisture barrier properties of polymer.

1.6.2 Application of crosslinked PVA film

- After improved moisture barrier properties of PVA film it can use in food packaging because PVA has good oxygen barrier property than other polymers.
- It can also use in pharmaceutical packaging to packaging of syringes, needles, bottles etc
- It can also use in FMCG product packaging like soap, detergent, shampoo, bakery items et

Chapter -2

Literature survey

Yun Zhang "crosslinking reaction of polyvinyl alcohol with glyoxal" In this paper they did crosslinking between polyvinyl alcohol and glutaraldehyde or glyoxal with the use of citric acid as a catalyst because PVA has hydroxyl group on their tail so it can easily react with aldehyde group. PVA is a completely biodegradable polymer easily soluble in water due to its hydrophilic nature. in this paper he reduced hydroxyl group from PVA and decreasing the crystallinity. Glutaraldehyde was chosen as crosslinker with pva because it favours the reaction between HCL and PVA. FTIR curve of this paper indicate the reduction of crystallinity due to crosslinking between PVA and glyoxal.

Role of boric acid for a poly vinyl alcohol film as a cross linking agent in this paper they were investigate the effect of aqueous boric acid on the dry pure PVA film they dipped the PVA film in aq. boric acid for cross linking at room temperature checked melting point and crystallinity of film after cross linking. They described that from this experiment crystallinity of PVA film were improved than before and melting point of PVA cross linked film increased. They type of characterization like TgA for finding melting point of PVA film and x-ray diffraction for checking crystallinity.

Crosslinking of polyvinyl alcohol with maleic anhydride by using electrospun. Electrospining PVA crosslinked with maleic anhydride to reduce water absorbing nature of PVA. maleic anhydride is slightly soluble in water its use as the crosslinker water solubility and melting point are described by different characterization. SEM described the morphology of electospun PVA and fibre structure of pure PVA and crosslinked PVA/MA. water durability test for water absorption and differential scanning calorimeter describes the crystal structure of PVA/MA.

Improvement of mechanical and thermal properties of polyvinyl alcohol by using DMSO. They used Hexamethyl diisocyanate solution for crosslinking between DMSO and PVA. And compared the mechanical and thermal properties of crosslinked PVA film with non crosslinked PVA film by using different type of characterization techniques. FTIR used for determine hydroxyl groups reduction and new bond formation of urethane compound .They did mechanical thermal analysis to check viscoelastic behaviour of crosslinked film and thermogravimetry test for measure thermal properties like melting point, residue point, time of evaporation etc.

Starch crosslinked with pva using hexamithoxyme thylmelamine as a crosslinker he studied about effect of mechanical, water absorption, and biodegradable properties on crosslinked cast starch/pva film or without crosslinked film. They found that crosslinked film absorb less water than uncrosslinked and moisture barrier properties of crosslinked also improved. He decided that by using characterization of film WVTR, Water absorption test, biodegradable test, and universal testing machine for check tensile strength of film.

For increase use of of biodegradable polymer in packaging they did crosslinking between polyvinyl alcohol and polycaprolacton in such a way PVOH losses their OH groups reduce their water solubility totally decomposed in soil. Its mechanical and physical properties defined by different characterization to determine bond they used FTIR and degree of crosslinked DSC test for the thermal properties defined. In the result they found that tg of pure pvoh is less than crosslinked film and melting point also increased'

Shude xiome did experiment of crosslinking between polyvinyl alcohol(PVOH) and trimesoyl chloride(TMC)/ hexane first he prepared pure pva film after drying he used TMC/ hexane at different concentration for crosslink after experiment they defined crosslinked by characterization FTIR, DSC, TGA, PERFOPERATION. They also determine degree of crosslinking.

Kwang zim kim described the effect of degree of crossslinked on pva crosslinked with glutaraldehyde by using different characterization FTIR, swelling test, contact angle and density measurement he desided that swelling of crosslink film reduce when we icrease degree of crosslinked and contact angle of film also change density will increase in ftir curve reduction of bend stretching.

Nadras Thomas described thermal properties of pvoh. he used blending between pvoh and corn starch casting method for film formation. He prepared different type of concentration film defined thermal properties by using TGA and DSC. From DSC graph desided that melting point of blend film decreased according to concentration of starch as we increased conc. Of starch in solution melting point, crystalline point and endothermic energy reduced continuously

Chapter -3

3.1 Experiment no.1- Formation of pure PVA film

3.1.1 Material required

Deionised water, polyvinyl alcohol, plastic plate, stirrer heater, magnetic bead, and beaker.

3.1.2 Method and preparation

Take 100 ml deionised water heated it upto 80°C 350rpm. Added 5g PVA(5% w/v) in heated water in five steps every step contain 1g PVA to dispersed. After this stirred solution for 3 hour at 80°C 350rpm. Prepared polymer solution poured in plastic plates. Keep all plate in oven for drying at 40°C for 24 hour. Dried films peeled carefully from casting surface.

3.1.3 Images of pure PVA film preparation

(a) (b) (c)

Fig:4(a)prepared solution of pure PVA (b) film before drying (c) film after drying

3.2 Experiment no.2: Formation of uncross linked PVA + Cinnamaldehyde film

3.2.1 Material required

Deionised water, polyvinyl alcohol, plastic plate, stirrer heater, magnetic bead, beaker, pipette, glycerol, and cinnamaldehyde

3.2.2 Method and preparation

Take 100 ml deionised water heated it upto 80°C 350 rpm. Added 5g PVA(5% w/v) in heated water in five steps every step contain 1g PVA to dispersed in water and stirred it for 3 hour at 80°C 350 rpm. after three hour reduced the temperature from 80°C to 40°C. Added 2ml cinnamaldehyde (2% of PVA solution) and glycerol (25% OF PVA) stirred the solution for two hour at 40°C 350 rpm. prepared solution poured into plastic plates all plates kept in the oven at 40°C for 24 hour. Dried films carefully peeled from casting surface.

3.2.3 Images of uncrosslink film preparation

(a) (b) (c)

Fig:5 (a)prepared solution for uncrosslink (b) film before drying (c) film after drying

3.3 Experiment no.3: Formation of cross linked PVA + Cinnamaldehyde film

3.3.1 Material required

Deionised water, polyvinyl alcohol, plastic plate, stirrer heater, magnetic bead, beaker, pipette, cinnamaldehyde, HCl, and glycerol.

3.3.2 Method and preparation

Take 100 ml deionised water heated it upto 80°C at 350 rpm. Added 5g PVA(5% w/v) in heated water in five steps every step contain 1g PVA to dispersed in water after this stirred it for 3 hour at 80°C 350 rpm. After three hour decreasing the temperature from 80°C to 40°C. added 2ml cinnamaldehyde(2% of PVA solution) stirred the solution for two hour at 40°C 350rpm. Added glycerol (25% of PVA) and maintain pH of solution by using hydrochloric acid.

3.3.3 Maintain pH

Required material:- pH meter, hydrochloric acid, distilled water

Before adding HCl in solution pH values

pH of HCl - 0.563

pH of solution - 4.56

pH of pH meter - 7.00

Added 2 drop of HCL in solution than pH of solution - 3.20

Again added 2 drop of HCL in solution - 2.56

Again added 2 drop of HCL in solution - 1.99

After maintain pH value at 2 stirred the solution for 3 hour at 40°C 350rpm. The prepared solution poured onto levelled plastic plate every plate dried at 40°C for 24 hour then the dried films carefully peeled from the casting surface.

Chapter- 4

4.1 Characterization

Colour test: The film colour were evaluated by an AWSC-S colorimeter we determined the parameters by placing the film sample on a standard plate. The lightness (L*) and chromaticity parameter [a*(red-green) and b*(yellow-blue)] were measured. The total colour difference (ΔE) were calculated with the following equation.

$$\Delta E= [(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]^{1/2}$$

FTIR: Fourier transform infrared spectroscopy is the instrument widely used in research and development to identification and quantization of organic solid, liquids, and gas samples. Analysis of powders, solids, gels, emulsion, pastes, polymers, and mix gases.. The cm^{-1} unit is the wave number scale. IR radiation causes the excitation and vibration within the molecule these vibrations include the stretching and bending modes. Energy absorption by the molecules cause of formation of spectral.

FESEM: The field emission scanning electron microscopy is on the principal of electron beam hits the sample, the interaction of beam electron from the filament and the sample atoms generate a variety of signals. Depending on the sample these can include secondary electron, backscattered electron, heat, light, and transmitted electron. SEM has an electron column where the electron are generated and arranged through electric and magnetic field a proper direction to achieve the required incident beam at the sample surface. The overall process is operated under vacuum to avoid collision between electrons and gas molecules. The SEM has different detector to view the electron signals coming from sample.

DSC: Differential scanning calorimetry is a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measure as a function of temperature both sample and reference are maintained at nearly the same temperature throughout the experiment. DSC used to study melting point of a crystalline polymer, glass transition temperature or exhothermic decompositions.

TGA: Thermogravimetric analysis is a method of thermal stability of material by using increasing temperature at constant rate of heat. We determine reduction of weight according with temperature increases continuously. Mass change of sample in overall TGA analysis in three steps in first step it tells about evaporation of water and stability of sample material in second step it gives the information about decomposition of sample (this is a slope line in graph) in third step it tells about the residue of material upto the highest temperature of graph. The graph is plot between weight percentage v/s temperature.

Water absorption test: it is a test for check total water absorb by material or a sample when we dip the sample in water it absorb water upto equilibrium stage the sample will swell after test. We have calculate percent of water absorb by sample at the equilibrium stage by using equation.

% of water absorption = (wet sample wt - dry sample wt)*100/ dry sample wt

WVTR: Water vapour transmission rate determine the permeation of vapour through the membrane this test conduct at particular relative humidity maintained in a chamber using desiccator solution.

Chapter - 5

5.1 Results and discussions

FTIR: The spectral analysis was determined by FTIR spectroscopy. KBr disks were used for FTIR measurement .The FTIR spectral analysis was performed at a resolution of 5cm^{-1} with 32 scans in the range 4500 to 500cm^{-1}.

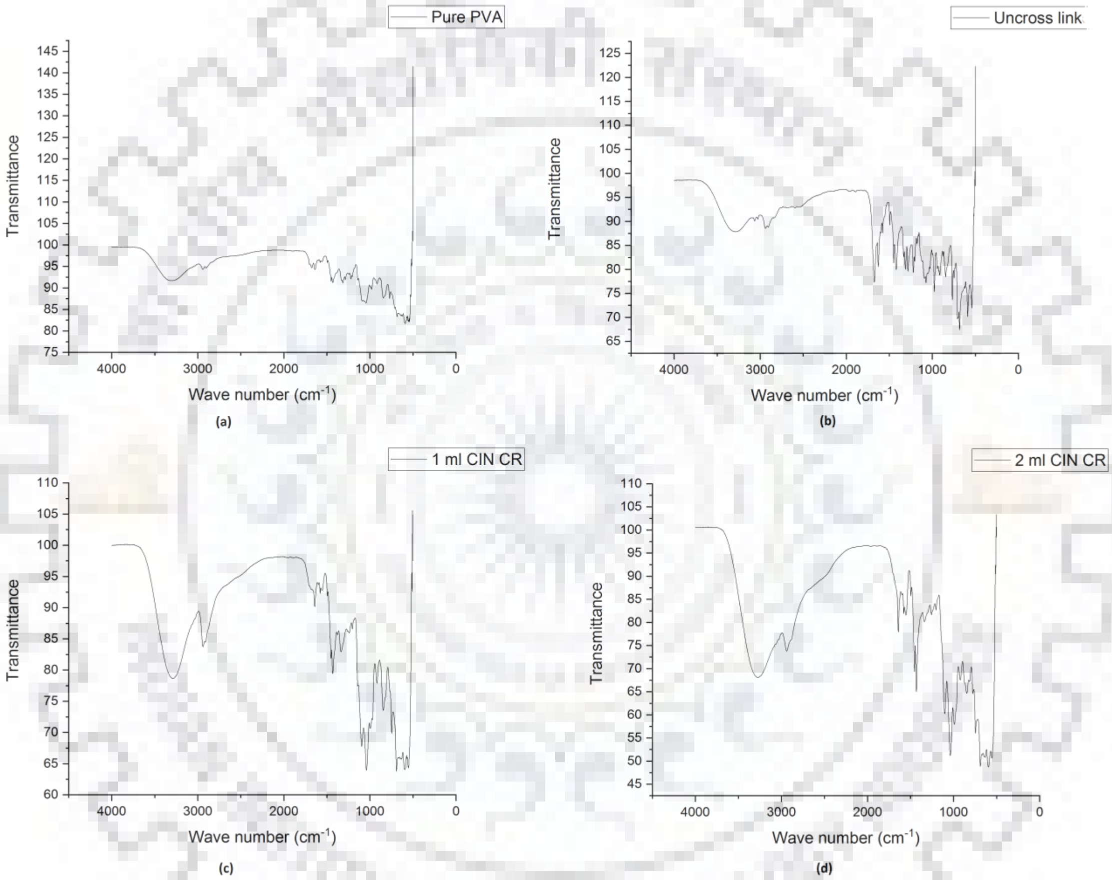


Fig:7 (a) pure pva (b)uncrosslink (c)1ml CIN crosslinked (d)2ml CIN

for the pristine PVA large bend between 3100 and 3600cm^{-1}were caused by O-H group. For the uncrosslinked PVA-CIN film shows the bands of PVA were at 2936 and 1424cm^{-1} which signifies to the bending and stretching of C-H and C-O in the C-O-H bond. The absorption peak at 2873 cm^{-1} signifies the C-H group of the aldehyde of CIN .For the crosslinked PVA-CIN film the formation of the new absorption peak at 980cm-1 corressponded to the stretching vibration of the C-O-C bonds of cyclic ether and C-H peaks. It disappeared at absorption peak of 2870 cm^{-1} of the aldehyde group of CIN as a result it shows the actualization occurred between the hydroxyl group of PVA and aldehyde group of CIN during the formation the crosslinking function. Due to the cross link between PVA and CIN the reduction in the intensity of the –OH bond also observed in the FT-IR spectra.

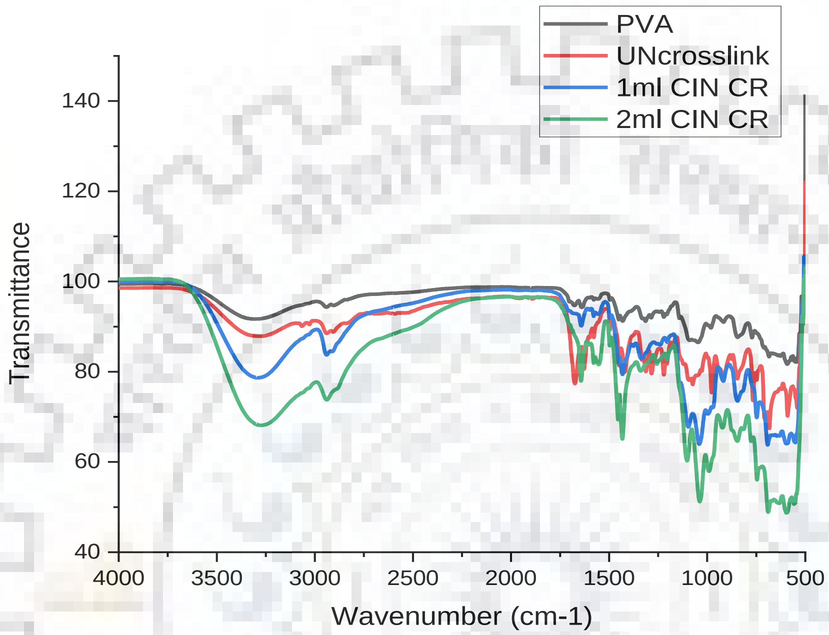


Fig:9.overall ftir graph for all samples

Chemical Reaction between polyvinyl alcohol and cinnamaldehyde in the presence of hydrochloric acid.

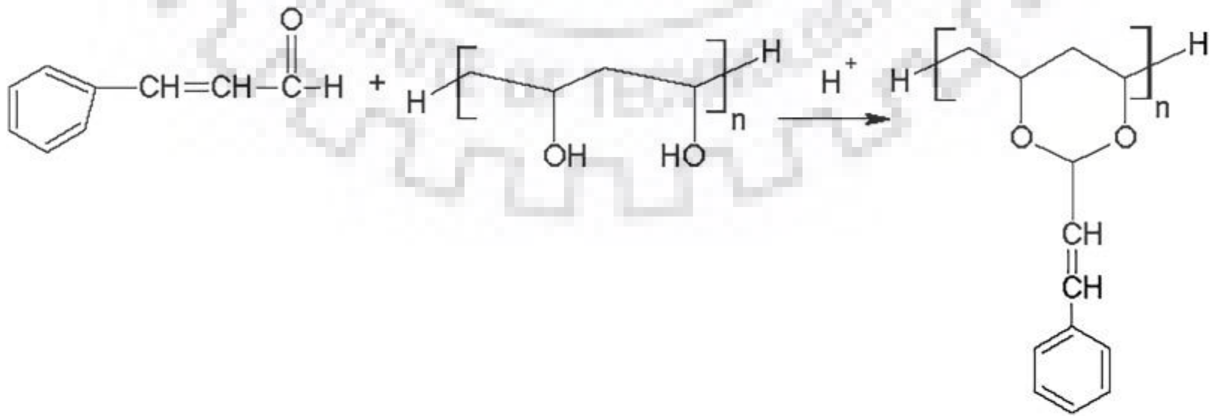
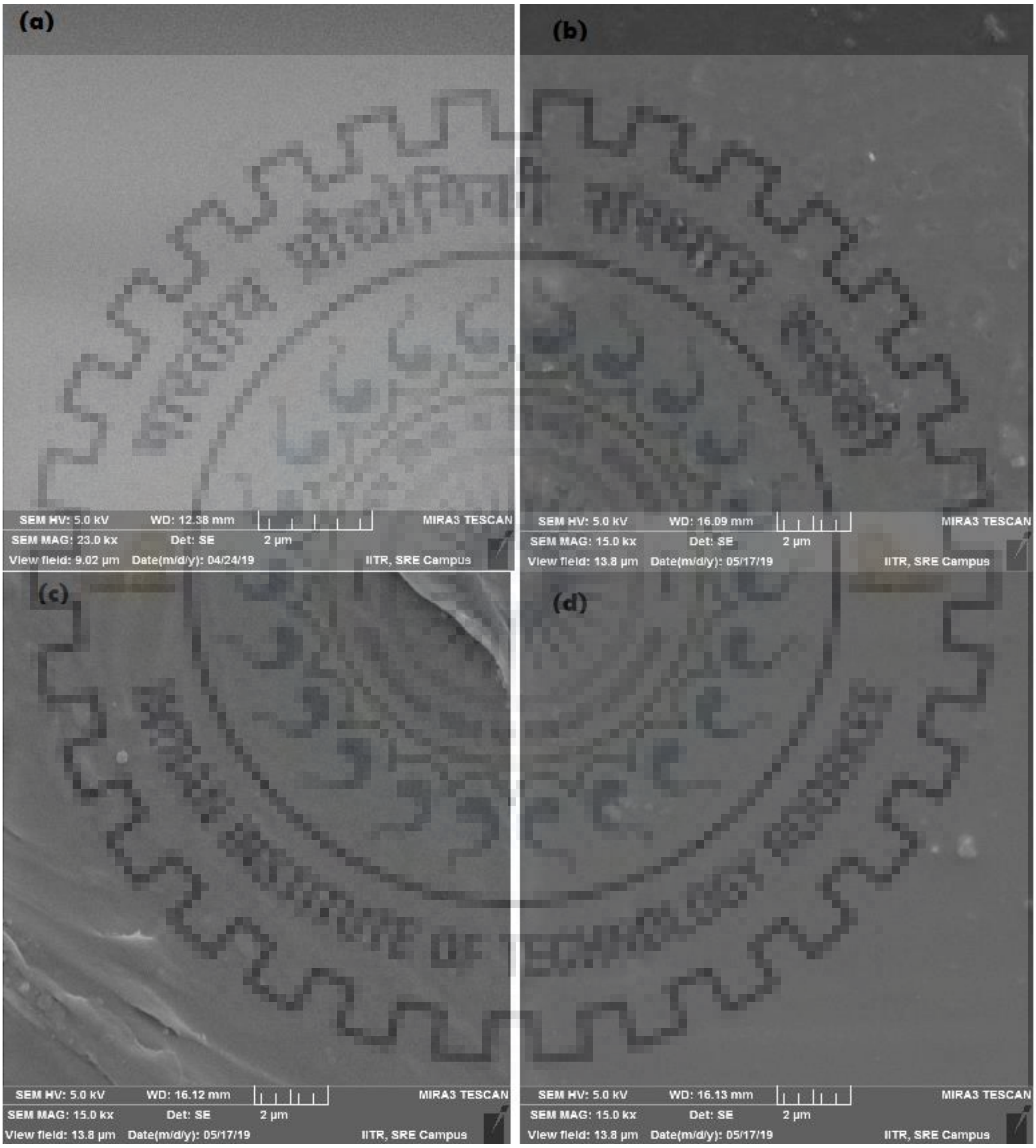


Fig:10. Chemical reaction between PVA and CIN

SEM:. The Film samples were coated with gold and observed at an accelerating voltage in the range of 1-5kV. The Pristine PVA film showed it to be smooth and even surfaces. In the uncrosslinked PVA-CIN film at lots of irregularities observed in the form of uneven surface and pore and cavities present in the film surface.



Colour test

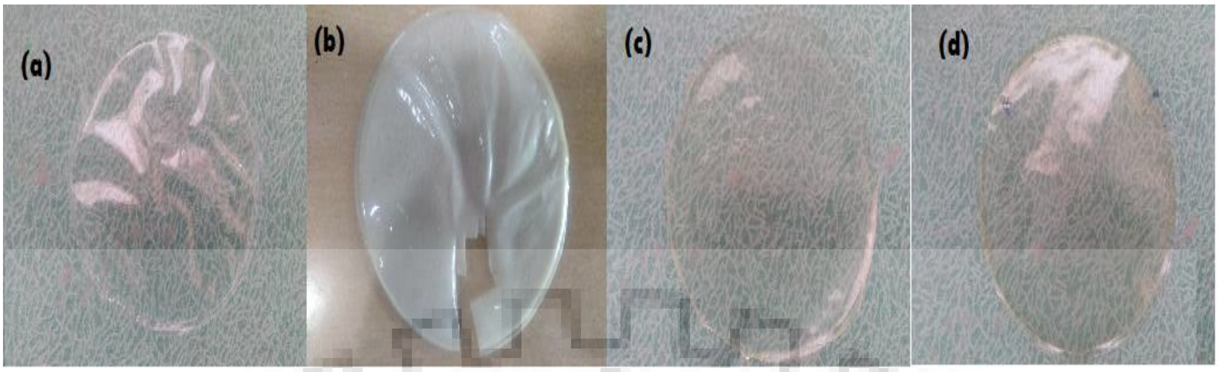
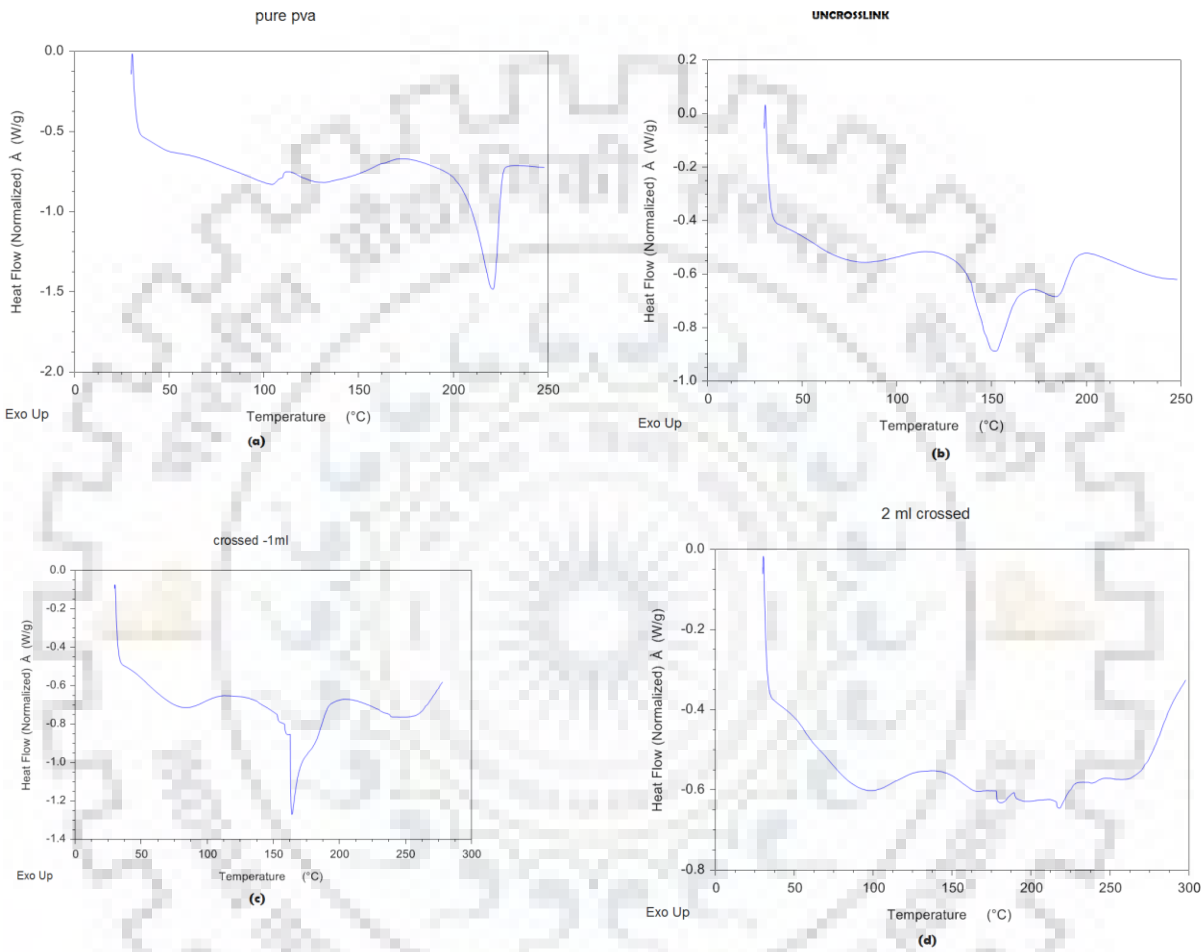


Fig:11 (a)pure PVA film (b)uncrosslink (c)1 ml CIN crosslink (d)2 ml CIN crosslink

Sample name	L^*	a*	b*	ΔE
Pure PVA	94.53	-0.22	0.15	2.28
Uncrosslink	94.49	-9.19	31.13	32.81
1ml CIN Cross	92.79	-1.24	5.16	6.86
2 ml CIN Cross	90.24	-1.45	9.77	12.69

From the above calculated data, Uncross linked PVA shows slightly increases in the lightness and drastically decrease in the greenish as well as the yellowish value as compared to pristine PVA. The total colour difference also increases for PVA as compared to Uncross-linked PVA. For cross-linked PVA-CIN film, when the concentration of CIN increases their is a decreases in lightness value, decreases in redness value and increase in yellowness value were observed.

DSC: This instrument technique is used to examile thermal behaviour of any polymer material. We are using it to examine behaviour of polyvinyl alcohol films in different concentration of CIN. We are studying four type of films pure PVA, uncrosslinked, 1ml CIN crosslinked, 2ml CIN crosslinked by using DSC maintain temperature range (0 °C to 300 °C) and heat flow rate 10°C/minute.



TGA: This is another thermal analysis technique to examine thermal properties of different kind of sample pure PVA, uncrosslink, 1ml CIN crosslinked, and 2ml CIN crosslinked at temperature range (0°C to 700°C) heating rate 10°C/min.

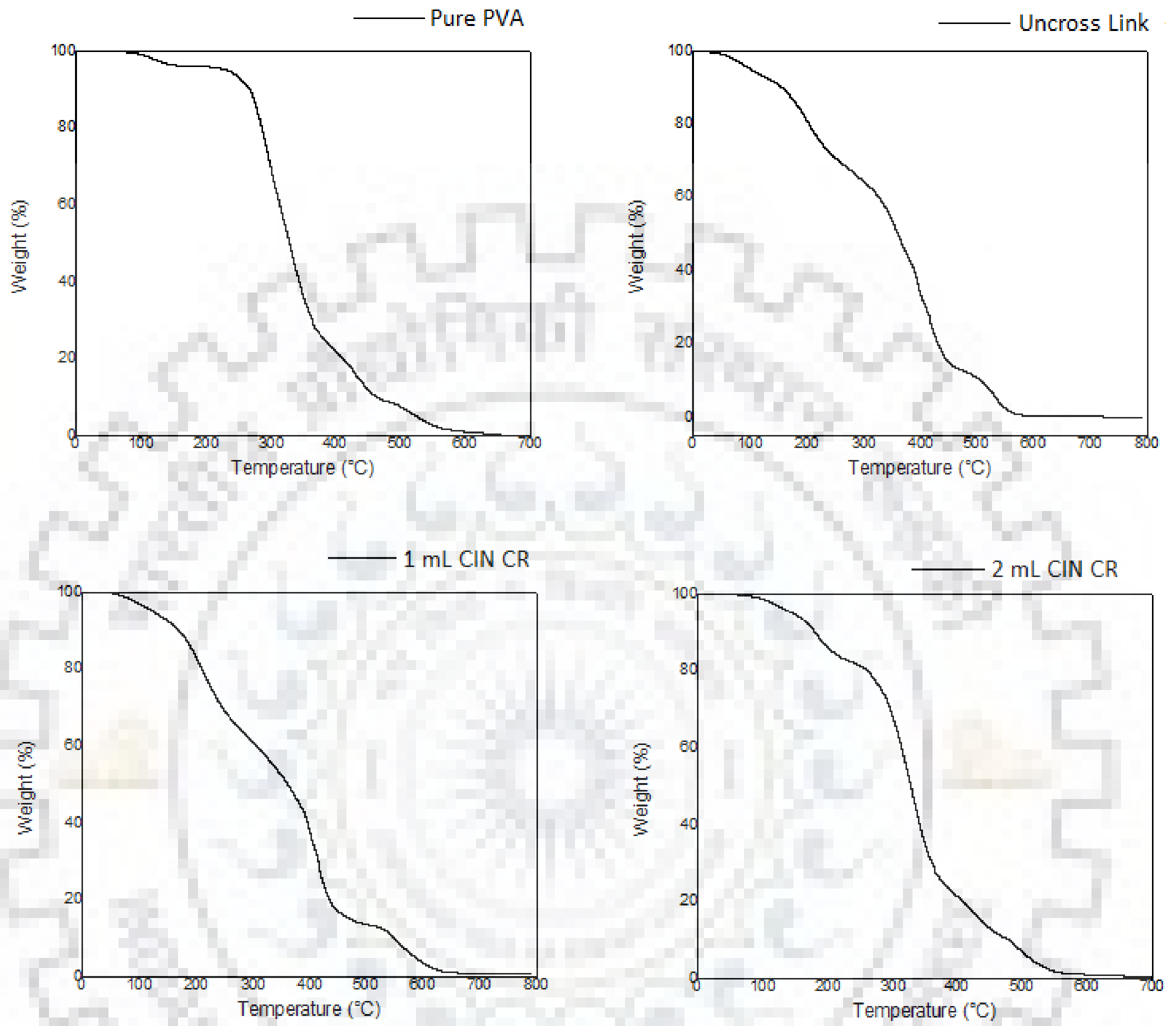


Fig:12 Thermogravimetric graph behaviour of different films

From the graph of pure PVA it is clear initially mass loss from temperature 80 to 220°C due to evaporation of water after this it shows inclined line from 220 to 400°C because of mass measure mass loss of PVA . if we talk about uncrosslink film initially mass loss start at 60 to 200°C after that its mass loss upto 420°C. For the crosslink film graph shows that initially mass loss is greater than pure PVA because presence of CIN ,glycerol and water. Main mass loss due to mass loss starts at 260°C due to crosslinking between CIN and PVA after all discussion it is clear that crosslinking reaction increased the melting point of PVA.

Water absorption test

Method followed ASTM –D570

For the water absorption test the specimen are dried in a oven for a specified time and then taken 150ml distilled water in beaker as per required of no. of beaker dip the specimen in water keep it for 24 hour at 23°C or until equilibrium.

The every specimen cuts in the circle form

Specimen diameter size - 50mm

% water absorption = (wet sample wt - dry sample wt)*100/ dry sample wt

Fig:13 film samples before water absorption test

Measured values

Sample name	Pure PVA	Uncrosslinked	Crosslinked 1 ml CIN	Crosslinked 2 ml CIN
Dry film	0.310	0.552	0.321	0.577
After 2 hour	1.535	1.947	0.518	0.664
After 4 hour	1.537	1.948	0.520	0.664
After 24 hour	1.540	1.960	0.521	0.664
After 48 hour	1.546	1.970	0.530	0.664
Total absorption of water	398.7%	256.8%	65.1%	15.07%

Fig:14. images of films after water absorption test

From the table it is clear that pure PVA sample specimen absorb more water than other three sample. Uncrosslinked film specimen absorb less water than pure PVA specimen due to CIN but absorb more water than other two sample specimen. crosslinked 2ml CIN specimen absorb very less water because of hydroxyl groups are removed by crosslinking reaction between PVA and CIN and alkoxy group are formed which has repellent nature against water

WVTR: water vapour transmission rate of film measured according to the **ASTM E 96-95** the sample were cut into round pieces and thickness were measured at six different point per sample. Then the tested films were sealed on top of a glass permeation cup the bottom of each cup filled with 10g silica gel. The cup holding films were then placed in a desiccators with 50% RH generated by saturated sodium bromide solution. The weight gain of silica gel were measured at 24 hour.

Sample name	Total initial wt of sample (gm)	Total final wt of sample after five days (gm)	Total moisture absorbed after 5 days(gm)	Average moisture absorbed per day (gm)	Calculated WVTR (gm/m^2day)
Pure PVA	62.535	62.619	0.084	0.0168	9.286
Uncrosslink	64.483	64.998	0.515	0.103	56.93
1ml CIN crosslinked	67.784	67.866	0.082	0.0164	9.065
2ml CIN crosslinked	63.352	63.415	0.063	0.0126	6.965

From the above table of result of WVTR it is clear that 2 ml crosslinked film showed excellent barrier against WVTR and very bed barrier showed by uncrooslink film because of essential oil dispersed into the polymer film due to this holes are created in films so water vapour easily pass thought it. Pure PVA has also quite good barrier properties against moisture pass.

Chapter - 6

CONCLUSION

After study of overall experiment and characterization it is clear that crosslinking of polyvinyl alcohol with cinnamaldehyde in the presence of hydrochloric acid in the favour of improvement of barrier properties of PVA crosslinked film. the improved barrier properties of polyvinyl alcohol crosslinked film indicate that it can be used in food packaging. But in overall experiment and characterization method it is not clear exactly how much CIN consume in crosslinked reaction and what is the effect coming due to presence of excess amount of CIN.

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