

SYNTHESIS AND CHARACTERIZATION OF CARBOXYMETHYL CELLULOSE / GLASS FIBER / POLYVINYL ALCOHOL MEMBRANES

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ABSTRACT

Ternary polymer blends were prepared by mixing of carboxymethyl cellulose, polyvinyl alcohol (PVA) and glass fibre in the various ratios. The characterization of various polymeric blends were carried out by advanced analytical techniques such as Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and Thermo gravimetric analysis (TGA). The changes in the degree of crystallinity of the prepared blend were elucidated from X-ray diffraction studies. From the TGA studies, it was evident that the prepared carboxymethyl cellulose /polyvinylalcohol/glass fibre has highly thermally stable behavior and the results were investigated.

KEYWORDS: Carboxymethyl cellulose, polyvinyl alcohol, glass

fibre, ternary blend.

INTRODUCTION

Blends of polymers represent class of materials with better mechanical properties and biocompatibility than those of single components.^[1,2] An important and determined aspect of the blend is the miscibility of the component. When two polymers undergo miscibility, well order micro structure results in giving the blend. Miscibility in polymer blends is assigned to specific interaction between polymers component, which is usually give rise to a negative free energy of mixing in spite of the high molecular weight of polymers.

Carboxymethyl Cellulose

Carboxymethyl cellulose was first produced in Germany in the 1930s. Since 1947, it was

manufactured in the USA.^[3] Carboxymethyl cellulose is a derivative of cellulose obtained by the chemical modification of natural cellulose. CMC is generally prepared through the reaction of alkali cellulose with mono chloro acetate or its sodium salt in an organic medium. Carboxymethyl cellulose has many desirable applications, such as in coating, the formation of emulsions and suspensions, and for water retention. Therefore it is used in many applications such as medicine, food, paper making, printing and dyeing.

Carboxy methyl cellulose is cellulose ether which displays thermal gelation and forms excellent films. Because of its polymeric structure and high molecular weight, it can be used as filler in bio-composite film production. Carboxy methyl cellulose can improve the mechanical and barrier properties of starch-based films.^[4] In addition, it is also used to improve the mechanical properties of the polymer by adding fillers. These composites could be used in packaging which is the good barrier and have the high thermal properties.^[5]

Polyvinyl Alcohol

Polyvinyl alcohol (CAS no 9002-89-5) was placed on the agenda of the 61st JECFA. Polyvinyl alcohol was first prepared by Hermann and Haehnel in 1924 by hydrolyzing polyvinyl acetate in ethanol with potassium hydroxide. Polyvinyl alcohol is produced commercially from polyvinyl acetate, usually by a continuous process. The acetate groups are hydrolyzed by ester interchange with methanol in the presence of anhydrous sodium methylate or aqueous sodium hydroxide.

The physical characteristics and its specific functional uses depend on the degree of polymerization and the degree of hydrolysis. Polyvinyl alcohol is an odorless and tasteless, translucent, white or cream colored granular powder. It is used as a moisture barrier film for food supplement tablets and for foods that contain inclusions or dry food with inclusions that need to be protected from moisture uptake.^[6] PVA is the most readily biodegradable of vinyl polymers. It is readily degraded in wastewater activated sludge. The excellent chemical resistance, optical and physical properties of PVA resins, has resulted in industrial uses.^{[7][8][9]} PVOH blends have a great interest according to their excellent compatibility and improved properties such as tensile strength, elongation, toughness and processability.^[10]

Glass Fiber

Ancient Egyptians made containers of coarse fibers drawn from heat softened glass. The French Scientist, Reaumur, considered the potential of forming fine glass fibers for woven

glass articles as early as the 18th century. Continuous glass fibers were first manufactured in substantial quantities by Owens Corning Textile Products in the 1930's for high temperature electrical applications.^[11]

Glass fibers are very useful in thermal insulators because of their high ratio of surface area to weight. However, the increased surface area makes them much more susceptible to chemical attack. By trapping air within them, blocks of glass fibers make good thermal insulation, with a thermal conductivity of the order of 0.05W/(c.m).^[12]

The glass fibers obtained as a solid residue in pyrolysis suffer the degradation of their physical properties, limiting their reuse.^[13] Raw materials such as silicates, soda, clay, limestone, boric acid, fluorspar or various metallic oxides are blended to form a glass batch which is melting in a furnace and refined during lateral flow to the earth.^[14]

MATERIALS AND METHODS

Materials

Carboxymethyl cellulose was provided by thermo fisher scientific private Ltd, Mumbai. Glass fibers which are made of silicon oxide with addition of small amounts of other oxides were purchased from supreme industries, Chennai. Polyvinyl alcohol was obtained from central Drug House Private Limited, New Delhi.

Methods

Preparation of Carboxymethyl Cellulose Solution

Carboxymethyl cellulose solution was prepared by dissolving a certain amount of CMC in minimum quantity of distilled water with constant stirring.

Preparation of Glass Fiber

Certain amount of glass fiber is dissolved in minimum quantity of distilled water with constant stirring.

Preparation of Polyvinyl Alcohol

A measured quantity of polyvinyl alcohol is dissolved in distilled water and it was then stirred for one hour.

RESULTS AND DISCUSSION

Fourier Transform Infrared (FTIR) Studies

Fourier transform infrared (FTIR) Spectra of Carboxymethyl cellulose (CMC)/ Glass fiber/ Polyvinyl alcohol (PVA) blends were performed with a Perkin Elmer 200 FT-IR spectrometer using KBr pellets. The FT-IR were obtained in the wave number range from 4000-350 cm^{-1} with a resolution of 4 cm^{-1} . In the FTIR spectrum of CMC (Figure 1), a broad band at 3360 cm^{-1} is due to the stretching of O-H group and a band at 2915 cm^{-1} due to the stretching of C-H group. However, a carbonyl (C=O) peak at 1590 cm^{-1} , a $-\text{CH}_2$ peak at 1414 cm^{-1} and an ether peak at 1046 cm^{-1} confirmed the introduction of a $-\text{OCH}_2\text{COO}$.^[15-17]

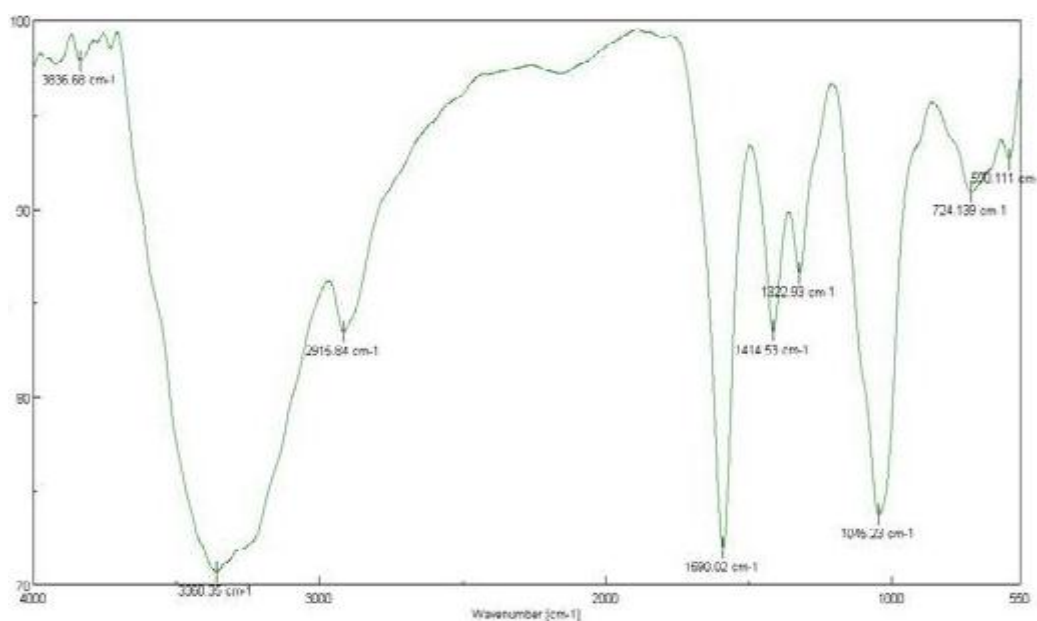


Figure 1: FTIR Spectrum of CMC.

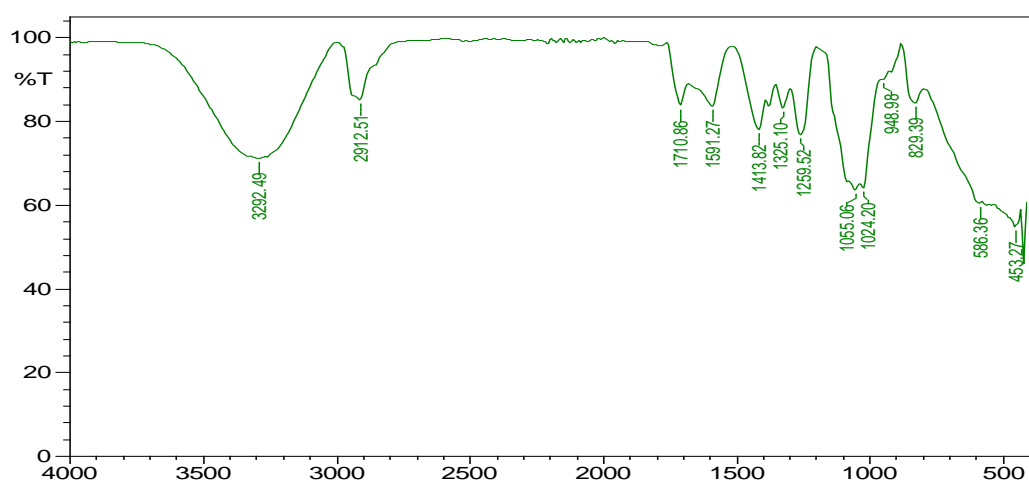


Figure 2: FTIR Spectra Studies Carboxymethyl Cellulose/Glass Fiber/ Polyvinyl Alcohol (1:1:1).

And Figure (2) represents the FT-IR spectral details of the Carboxymethyl cellulose/Glass fiber/Polyvinyl Alcohol (1:1:1) membrane. The band observed at 3292.49 cm^{-1} , 2912.51 cm^{-1} , indicate the stretching vibration of intermolecular hydrogen bonded -OH stretching of axial OH group, C-H stretching in CH_2 , C=O stretching, C-H bending, C-(C=O)-O stretching in esters C-O stretching in alcohols C-H plane bending 1710.86 cm^{-1} , 1413.82 cm^{-1} , 1259.52 cm^{-1} , 1024.20 cm^{-1} 948.98 cm^{-1} respectively. Certain peaks which was obtained at 897.97 cm^{-1} 453.27 cm^{-1} was attributed Si-O vibration and C-C bending vibration.

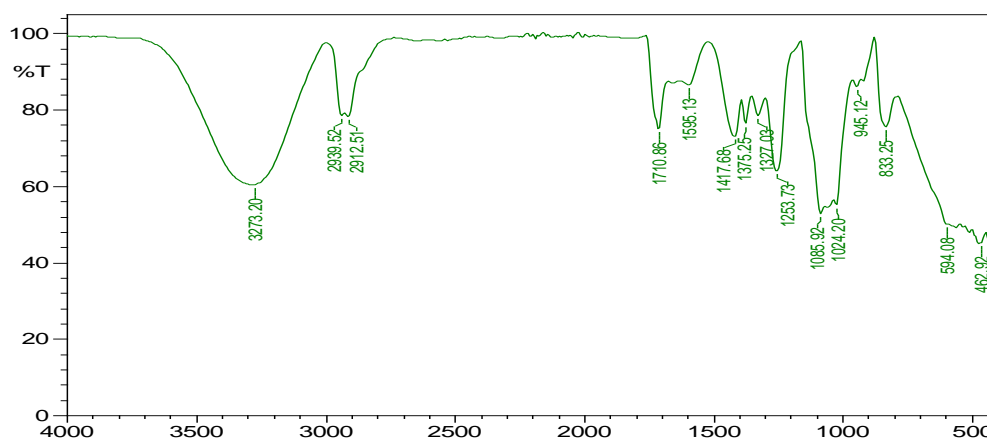


Figure 3: FTIR Spectra Studies Carboxymethyl Cellulose/Glass Fiber/ Polyvinyl Alcohol (1:1:2).

The FT-IR spectra details of the Carboxymethyl cellulose/Glass fiber/Polyvinyl Alcohol (1:1:2) membrane was shown in the figure (3). The prominent peak observed at 3273.20 cm^{-1} , 2912.51 cm^{-1} , 1710.86 cm^{-1} , was due to the intermolecular hydrogen bonded -OH stretching, aliphatic -CH stretching and C=O stretching respectively. Certain peaks observed at 1417.68 cm^{-1} , 1253.73 cm^{-1} , 1024.20 cm^{-1} was attributed to C-H bending, C-(C=O)-O stretching in esters and C-O stretching. The peak obtained at 945.12 cm^{-1} , 833.25 cm^{-1} , 462.92 cm^{-1} was due to the C-H plane bending, Si-O vibration and C-C bending.

While comparing the FT-IR spectral details of Carboxymethyl cellulose/Glass fiber/Polyvinyl Alcohol membrane prepared in two ratios with pure polyvinyl alcohol it was observed that so many additional bands were obtained at various wave number such as 3273.20 cm^{-1} , 2912.51 cm^{-1} which corresponds to OH stretching and aliphatic C-H stretching. The above observed additional peak confirms that the blending had taken place effectively between Carboxymethyl cellulose, Glass fiber and Polyvinyl Alcohol.^[18]

X-Ray Diffraction (XRD) Studies

x-ray diffraction (XRD) patterns of CMC/Glass fiber/PVA ternary blends samples were tested by an X-ray scattering Shimadzu diffractometer using Ni filtered Cu K α radiation source (0.15nm), set at a scan rate of 10mins using a voltage of 40kv and a current of 30milliamperes.

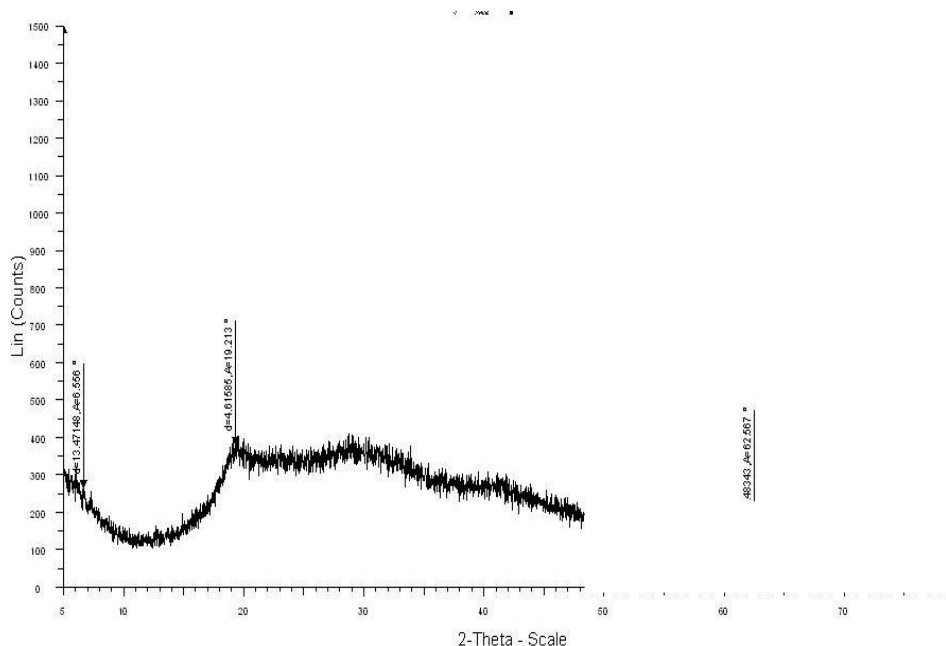


Figure 4: XRD Spectra Studies Carboxymethyl Cellulose/Glass Fiber/ Polyvinyl Alcohol (1:1:1).

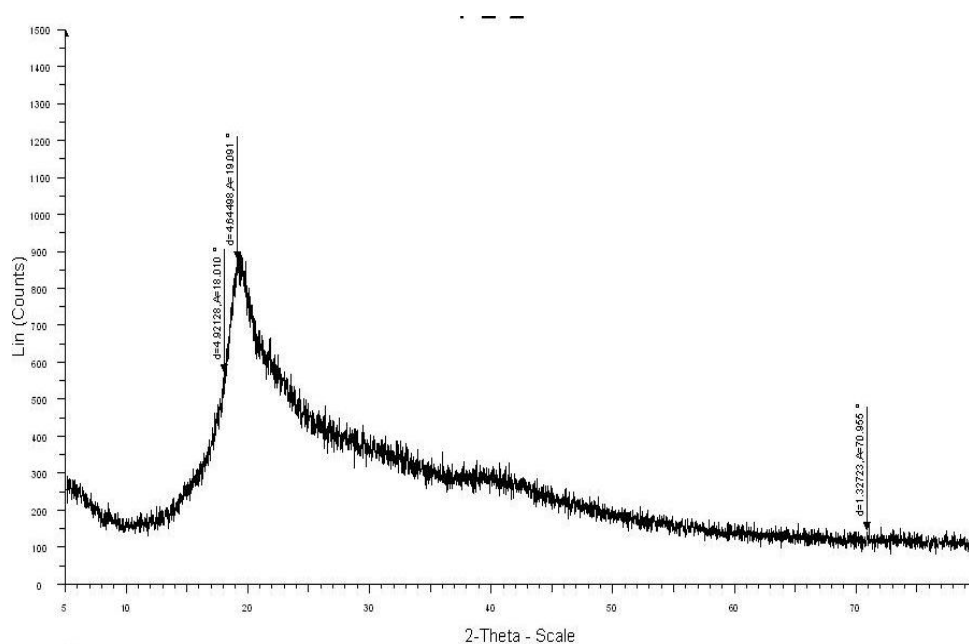


Figure 5: XRD Spectra Studies Carboxymethyl Cellulose/Glass Fiber/ Polyvinyl Alcohol (1:1:2).

Crystallinity

The degree of crystallinity of a blend polymer measures the ratio of crystalline part to the amorphous part, when two semi crystalline polymers are mixed.

The degree of crystallinity value observed for the blends prepared under the various conditions are represented in the table and figure.

It was observed that the membranes prepared in various ratios using the carboxymethyl cellulose, glass fiber and polyvinyl alcohol shows the peaks at various 2-theta values such as 6°, 19°, 62°, 19°, 20°, 71°. The broad nature of the peak and the lower percentage of crystallinity values observed in the case of the carboxymethyl cellulose/glass fiber/polyvinyl alcohol prepared in (1:1:1) ratio when compared to the membranes prepared in other ratio. This indicate that the membrane M₁ (1:1:1) is more amorphous than the other ratio which may be due to hydrogen bond formation and greater miscibility. In addition to this, the above results also prove that the blending takes place effectively between Carboxymethyl cellulose / Glass fiber / Polyvinyl Alcohol membranes.

Thermo Gravimetric Analysis Studies of the Blends (TGA)

Thermo gravimetric analysis of ternary blends prepared using CMC/Glass fiber/PVA blends was done using Perkin Elmer thermal analysis instrument. The temperature range was varied from 35°C-850°C with the heating rate of 100°C/min. In this technique the change in mass of the substance was measured as a function of temperature. The DTG curves of the prepared blends exhibited three distinct weight loss stages. The first stage of degradation represent the volatilization of easily degraded components, while the second stage exhibits the major mass loss predominantly the characteristic degradation of polymer structure (water elimination of PVA, decomposition of starch and decomposition CMC). The third stage of weight loss shows the decomposition of main chain of PVA.^[19-22]

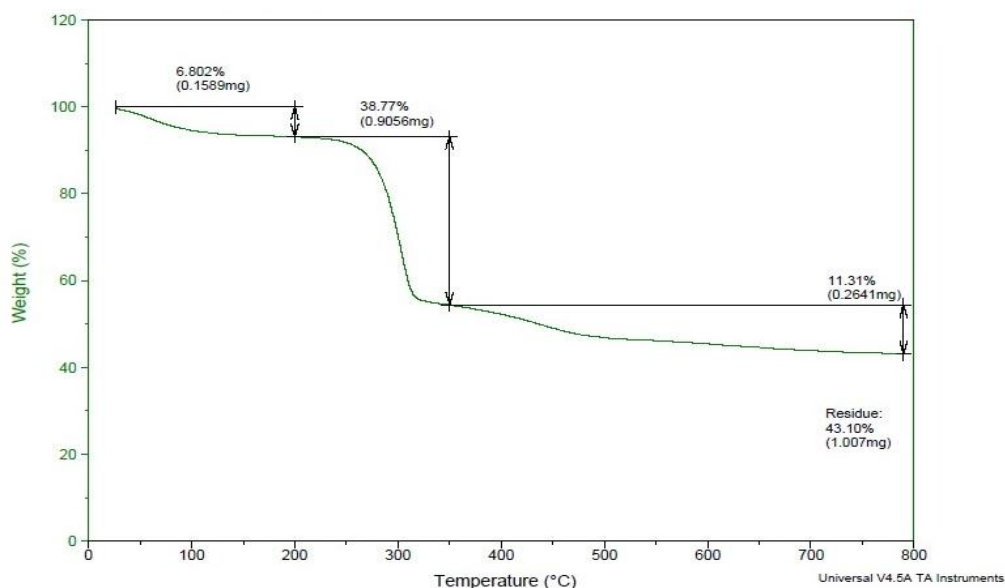


Figure 6: TGA Spectra Studies Carboxymethyl Cellulose/Glass Fiber/ Polyvinyl Alcohol (1:1:1).

The amount of residue indicates that the significant amount of organic materials is retained. From the comparison of TGA results of the membranes prepared in various proportions it was concluded that the carboxymethyl cellulose/ glass fiber / polyvinyl alcohol (1:1:1) membrane is thermally more stable. This was confirmed from the amount of residue remained at the end of the experiment and various degradation temperature. In addition to this it was also observed Carboxymethyl cellulose increases the thermal stability of glass fiber, slowing down its thermal degradation.^[23]

CONCLUSION

The Carboxymethyl cellulose (CMC)/ Glass fiber/ Polyvinyl alcohol (PVA) membrane were successfully prepared in two different ratio. From the FT-IR Spectra result it is prominently observed certain peaks are obtained due to the intermolecular hydrogen bonded –OH stretching, aliphatic –CH stretching respectively. So it suggested that the strong interaction takes place between the ternary blend. The X-ray diffraction (XRD) studies reveals the nature of the peak and the lower percentage of crystallinity values observed in the case of the carboxymethyl cellulose/glass fiber/polyvinyl alcohol prepared in (1:1:1) ratio when compared to the membranes prepared in other ratio. From the comparison of TGA results of the membranes prepared in various proportions it was concluded that the carboxymethyl cellulose/ glass fiber / polyvinyl alcohol (1:1:1) membrane is thermally more stable.

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REFERENCES

1. U. W. Gedde. “*Polymer Physics*” Chapman & Hall Ch., 1995; 4.
2. C. Uma Devi, A.K. Sharma and V.V.R. N.Rao, *Materials letters*, 2002; 56: 167.
3. Karolina Boruvkova, Jakub Wiener, Michaels Jakubickov. Preparation and properties of microporous structure based on CMC, 2011.
4. Ma X, Chang PR, Yu J, Properties of biodegradable thermoplastic pea starch/carboxymethyl cellulose and pea starch/microcrystalline cellulose composites. *Carbohydr Polym*, 2008; 72: 369–375.
5. Taghizadeh MT, Abbasi Z, Nasrolahzadeh Z, Study of enzymatic degradation and water absorption of nanocomposites starch/ polyvinyl alcohol and sodium montmorillonite clay. *J Taiwan Institute Chem Engine*, 2011; 43: 120–124.
6. S.K.Saxena Polyvinyl alcohol (PVA). Chemical and Technical assessment (CTA), First Draft, 2004.
7. Suzuki T, Degradation of poly(vinyl alcohol) by microorganisms. *J Appl Polym Sci Appl Polym Symposium*, 1979; 35: 431–437.
8. Watanabe Y, Morita M, Hamada N, Tsujisaka Y, Formation of hydrogen peroxide by a polyvinyl alcohol degrading enzyme. *Agric Biol Chem.*, 1975; 39: 2447–2448.
9. Holland B. J, Hay N, *Polymer*, 2001; 42: 6775- 6783.
10. Morita M, Watanabe Y, A secondary alcohol oxidase: a component of a polyvinyl alcohol degrading enzyme preparation. *Agric Biol Chem.*, 1977; 41: 1535–1537.
11. Taghizadeh MT, Mehrdad A Calculation of the rate constant for the ultrasonic degradation of aqueous solutions of polyvinyl alcohol by viscometry. *Ultrason Sonochem*, 2003; 10: 309–313.
12. W. W. Wolf. “The Glass Fiber Industry- The Reason for the Use of Certain Chemical Compositions”, Seminar at University of Illinois, Urbana, IL, 1982.
13. Frank P. Incropera; David. P.De Witt, 1990.
14. S. J. Pickering, “Recycling Technologies for Thermoset Composite Materials-Current Status,” *Composite Part A*, 2006; 37(8): 1206-1215.
15. P.K. Gupta, “Examination of the Textile Strength of E-Glass Fiber in the Context of Slow Crack Growth Fracture Mechanics of Ceramics” 1983; 5: 291.

16. Konto T, Cellulose, 1997; 4: 60-63.
17. Rachtanpun P, Kumthai S, Yagi N, Uthaiyod N, Kasetsart University Annual Conference, Proceeding, 2007; 790- 799.
18. Rachtanpun P, Mulkarat N, Pintajam N, The 5th International Packaging Congress and Exhibition, Turkey, 2007; 22-24.
19. Bella G.R., Jeba Jeevitha R, polyvinylalcohol/starch/carboxymethyl cellulose ternary polymer blends: Synthesis, Characterization and Thermal properties, 2016; 3. - ISSN:2348-5213: e-2348-5221.
20. Holland B. J, Hay N, Polymer, 2001; 42: 6775- 6783.
21. Thomas P. S, Guerbois J.P, Rueeell G. F, Briscose B. J, Journal of Thermal analysis and Calorimetry, 2001; 64: 501-508.
22. Vega D, Villar M.A, Failla M. D, Valles V.M, Polymer Bulletin, 1996; 37: 229-235.
23. Zhou X. Y, Jia D.M, Cui Y. F, Xie D, Journal of Reinforced Plastics and Composites, 2009; 28(22): 2771-2780.
24. S. El-Sayed b, n, K. H. Mahmoud, A. A. Fatah, A. Hassen. DSC, TGA and dielectric properties of carboxymethylcellulose/polyvinyl alcohol blends, 2011.