International Journal of Enhanced Research in Science Technology & Engineering, ISSN: 2319-7463
Vol. 4 Issue 4, April-2015, pp: (172-180), Impact Factor: 1.252, Available online at: www.erpublications.com

Fourier Transform Infra Red (FTIR) Spectroscopy of New Copolymers of Acrylic Resin Denture Base Materials

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ABSTRACT

Aims: Analyze and characterize the structure of new copolymers by Fourier Transform Infra Red (FTIR) spectroscopy.

Materials & Methods: Specimens of new copolymers were made by adding Meth Acrylic Acid (MAA), Butyl Meth Acrylate (BMA), and Ethyl Acrylate (EA) with 5%, 10%, and 15% concentrations to Vertex heat cured acrylic resin. FTIR spectra of the specimens were recorded by CL Alpha–P FTIR spectroscopy and viewed on computer device.

Results: The C=C band from the methacrylate group disappeared. A new band appeared at 2358 cm^{-1}. The remaining stretching vibrations are similar to the control group.

Conclusions: The modification of heat cured acrylic resin denture base material resulted in the formation of new copolymers.

Keywords: FTIR, copolymers, acrylic resin.

INTRODUCTION

Infrared spectroscopy may be used to identify the composition of polymers, to monitor polymerization processes, to characterize polymer structure, to examine polymer surfaces and to investigate polymer degradation processes.[1]

Fourier Transform Infrared Spectroscopy (FTIR) is one of the most widely used and well-established spectroscopic methods for analyzing and characterize the structure of polymers. FTIR spectroscopy present a sensitive analysis tool to detect composition changes in biomaterials.[2]

Copolymer is a polymer derived from two (or more) monomeric species, as opposed to a homopolymer where only one monomer is used.[3]

Copolymerization refers to methods used to chemically synthesize a copolymer. Copolymers may also be described in terms of the existence of or arrangement of branches in the polymer structure.[4]

Recently, there have been many materials used for denture base such as polymethyl methacrylate (PMMA) resin, modified PMMA resin and nylon. The material most often used to fabricate denture base and denture teeth was PMMA resin. PMMA is the most commonly used material due to its good mechanical and physical properties, compatibility with oral tissue, aesthetics, ease of repair and low cost. However, some problems such as denture fracture and wear of the denture teeth still exist. In order to overcome these problems, several attempts were made to modify and improve them Meth Acrylic Acid (MAA), is an organic compound. This colourless, viscous liquid is a carboxylic acid with a characteristic odor. It is soluble in warm water and miscible with most organic solvents. Meth Acrylic Acid is produced industrially on a large scale as a precursor to its esters, especially methyl methacrylate (MMA) and poly(methyl methacrylate) (PMMA). The methacrylates have numerous uses, most notably in the manufacture of polymers. Meth Acrylic Acid occurs naturally in small amounts in the oil of Roman chamomile.[8]

Butyl Meth Acrylate (BMA) are produced for the use as a building block to make a wide range of polymer based products that we see and use every day from paints and coatings, toners and inks, oil additives to dental and medical products. Butyl Meth Acrylate are of low concern to human health and the environment and have been handled safely

by industry and professionals for over 60 years. Butyl Meth Acrylate based polymers is inert in the environment and can be recycled back to the monomer. Butyl Meth Acrylate is produced for the use as monomer for production of polymers.[9]

Ethyl Acrylate (EA) is an organic compound. It is the ethylester of acrylic acid. It is a colourless liquid with a characteristic odor.[10]

Ethyl Acrylate is used in the production of polymers including resins, plastics, rubber, and denture material.[11]

The aims of the study are to analyze and characterize the structure of new copolymers(the modification of heat cured acrylic resin denture base material through the addition of: Meth Acrylic Acid, Butyl Meth Acrylate, and Ethyl Acrylate using three different concentrations;5%, 10%, and 15%) by Fourier Transform Infra Red (FTIR) spectroscopy.

MATERIALS & METHODS

The specimens were prepared from (Vertex™, Netherlands) heat cured denture base acrylic resin material with additives. The three types of acrylate derivatives additives are: Meth Acrylic Acid (MAA), Butyl Meth Acrylate (BMA), and Ethyl Acrylate (EA). All additives are manufactured by (Fluka, Switzerland) chemical industries. The additive materials have been added to the monomer of acrylic at a percentage of 5%, 10%, and 15%. The control specimens were prepared from (Vertex™, Netherlands) heat cured acrylic resin material without additives. For each variable group, first, the additive was added to monomer, and mixed together until a homogeneous mixture liquid was obtained. Then, the powder was added to the liquid according to the manufacturer instructions ratio (22g powder/10ml monomer).

The powder and liquid was mixed, covered, and waited 30 minutes to reach dough stage. The dough, then, applied into the molds and pressed to 200 μpa pressure for 10 minutes, then cured in short cycle according to manufacturer instructions (start with tap water and gradually increase the temperature until reach boiling degree and maintain at 100^{o}C for 30 minutes, bench cooling then open the flask). The Fourier transform infrared spectroscopy (FTIR) was carried by CL Alpha–P FTIR spectroscopy (Bruker Germany). Ten specimens were prepared for FTIR test with dimensions of 10x4x4 (± 0.03) mm (length, width, and thickness respectively).[12]One specimen for each group as follows:

Acrylic resin without additive (control)., Acrylic resin with 5% MAA.,. Acrylic resin with 10% MAA., Acrylic resin with 15% MAA.,. Acrylic resin with 5% BMA., Acrylic resin with 10% BMA., Acrylic resin with 15% BMA., Acrylic resin with 5% EA., Acrylic resin with 10% EA., Acrylic resin with 15% EA. After preparation of the specimens, they were finished with stone bur and sand paper to remove any remnants of gypsum products from their surfaces, then they were stored for 48 hour in distilled water at 37(±1)ºC.[12]

After 48 hour, the specimens were removed and dried in air for 24 hour to remove the water from the specimens, and then, the dried resins were scraped by using sharp sterile wax knife to form small pieces of acrylic resin to obtain a clear FTIR spectrum. Then a1 – 2 mg polymerized powder is placed in the sample beam of the spectrometer. The specimens were placed on the diamond set, and FTIR spectra of the specimens were recorded and viewed on the computer device.[13]

RESULTS

Figure (1) showed the FTIR chart of the control group (heat cured acrylic resin without additive), The FTIR spectra shows the main expected bands characterizing the vibrational spectrum of PMMA, The band at 3000 cm^{-1} is assigned to CH_3 stretch vibration,The characteristic methylene C–H stretches bands is at 2949 cm^{-1},.The ester carbonyl C=O stretching vibrations are at 1731–1718 cm^{-1},The carbon-to-carbon double bonds (C=C) concentration of the uncured material(from the methacrylate group) are at1684– 1636 cm^{-1}**,**The CH_2 aromatic group is at the band 1437 cm^{-1}, The characteristic broad peak ranging from 1260–1000cm^{-1} can be explained owing to the C–O (ester band) stretching vibration,The C–O–C vibration is at 1141 cm^{-1}**,** The broad band from 950–650 cm^{-1} is due to the bending of C–H.

Figures (2,3,4) showed the FTIR charts of the acrylic resin with 5%MAA,10%MAA, and 15%MAA respectively. There are two differences between them and the control group. The first is that the C=C band from the methacrylate group disappeared. The second is that a new band appeared at 2358 cm^{-1}. The remaining stretching vibrations are similar to the control group. The new band confirms that a new structure of polymer had been formed.

Figures (5,6,7) showed the FTIR charts of the acrylic resin with 5%BMA, 10%BMA, and 15%BMA respectively. The C=C band from the methacrylate group disappeared. A new band (not present in the control group) appeared at 2358

cm^{-1}. This new band appeared more stretched than that appeared in the MAA groups. The stretching of the new band increased with the increase of the concentration of the BMA material. The remaining bands of the BMA groups are similar to the control group (acrylic resin without additives). The new band confirms that a new structure of polymer had been formed.

Figures (8,9,10) showed the FTIR charts of the acrylic resin with 5%EA, 10%EA, and 15%EA respectively. The C=C band from the methacrylate group disappeared. A new band appeared at 2358 cm^{-1} (not present in the control group).This new band appeared more stretched than that appeared in the MAA groups. The stretching of the new band is the same in the three concentration of the EA material. The remaining vibrational bands of the EA groups are not changed in comparison with the control group. The new band confirms that a new structure of polymer had been formed.

DISCUSSION

Vibration spectroscopy has been utilized for the characterization of polymers and other material. Variations in the environment of molecular components of materials are reflected in shifts in absorbance band intensities and positions in the vibration spectra. The spectrum of a material provides insight into the chemical composition of absorbance and how it might be altered during processing.[14]

Fourier transform infrared spectroscopy (FTIR) is a powerful analytical technique that has been utilized as a quantitative measure for the identification and monitoring setting reactions and polymerization of a broad range of dental materials.[15]

During curing of acrylic resin, polymerization is initiated by free radicals from the benzoyl peroxide. As polymerization proceeds, the reaction never reach 100% conversion. i.e. conversion of monomer into polymer is not completed. The degree of conversion is the most important criterion that account for unreacted residual monomer levels.[16]

The degree of conversion is expressed as percentage of unreacted C=C bonds.[17]

The degree of conversion of dimethacrynlates may be improved if the distance between the methacrylate groups is long, and the molecular weight is high, respectively. High conversion is not a goal in itself, however, if the monomer is very flexible, and not sufficiently bulky, the degree of conversion will be high, but the mechanical properties will be poor.[18]The final degree of conversion of a resin depends on the chemical structure of the dimethacrylate monomer and the polymerization conditions. i.e., atmosphere, temperature, light intensity and photo initiator concentration.[12]

CONCLUSION

The modification of heat cured acrylic resin denture base material (through the addition of: Meth Acrylic Acid, Butyl Meth Acrylate, and Ethyl Acrylate using three different concentrations; 5%, 10%, and 15%) result in the formation of new copolymers.

AKNOWLEDGMENT

Special thanks for Dr. Amer A. Taqa, Professor of Chemistry, College of Dentistry, University of Mosul, for his help and support.

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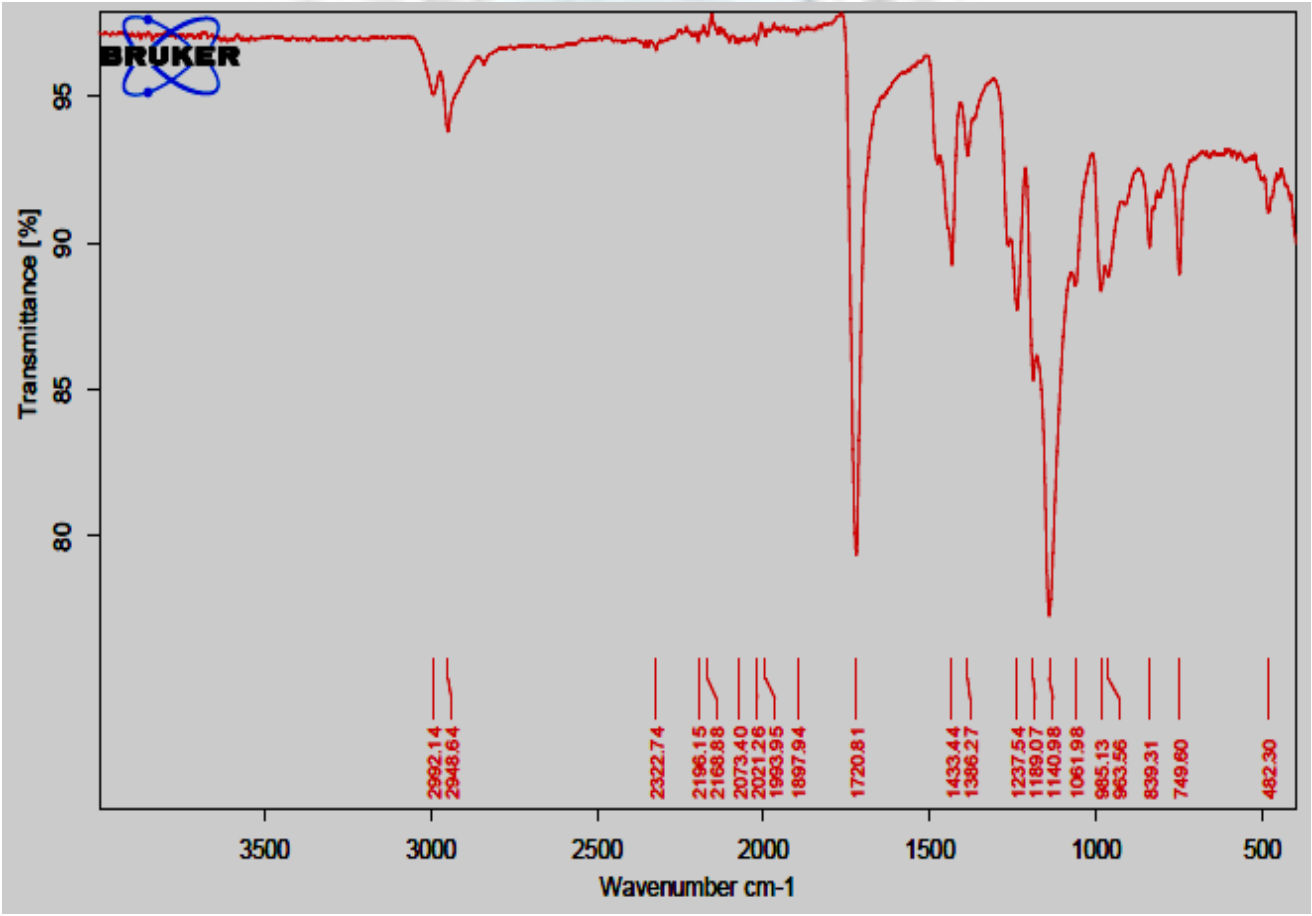

Figure (1): FTIR for control (heat cured acrylic resin without additive)

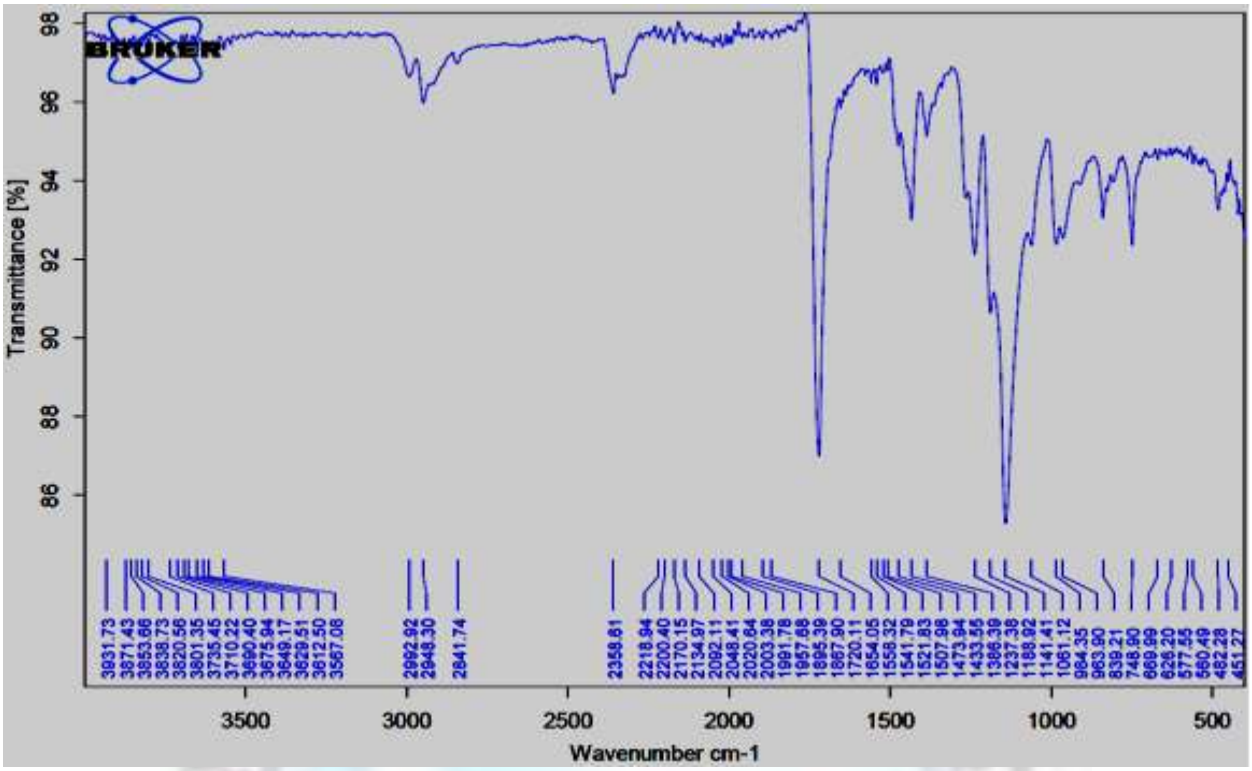

Figure (2): FTIR for 5% MAA

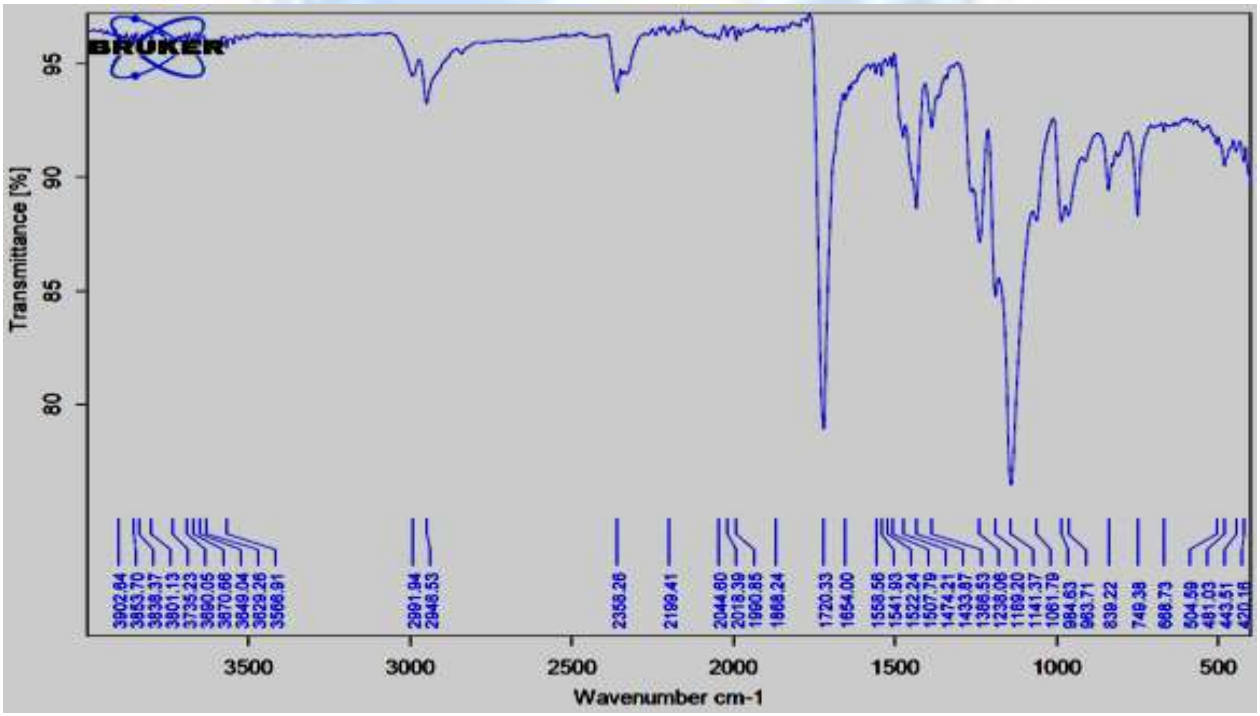

Figure (3): FTIR for 10% MAA

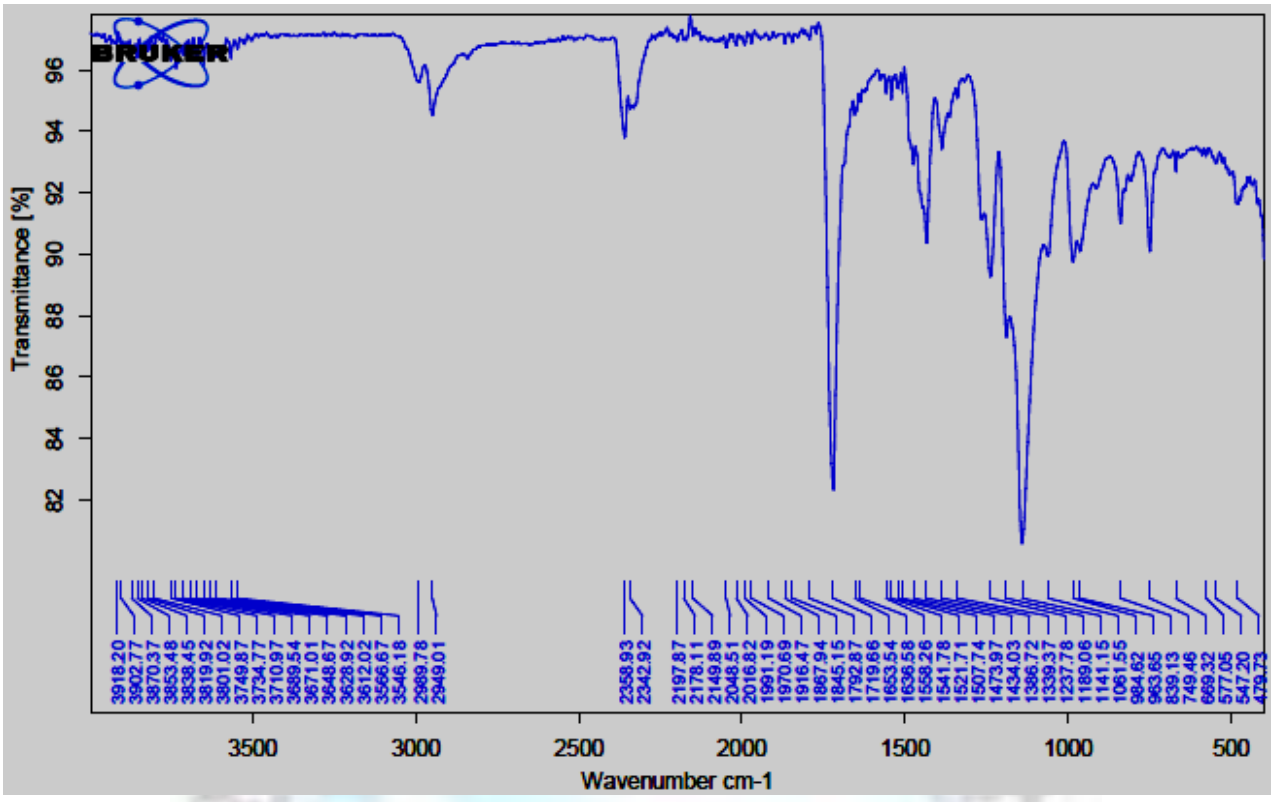

Figure (4): FTIR for 15% MAA

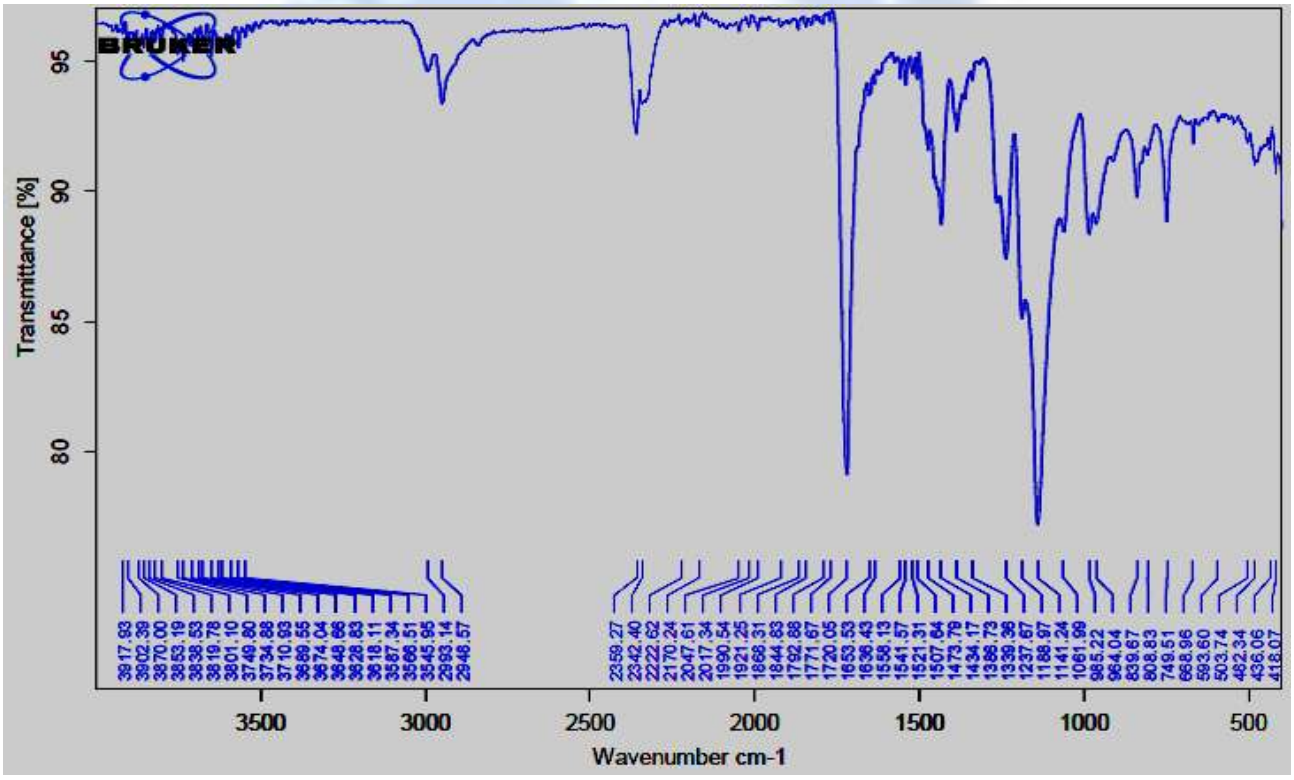

Figure (5): FTIR for 5% BMA

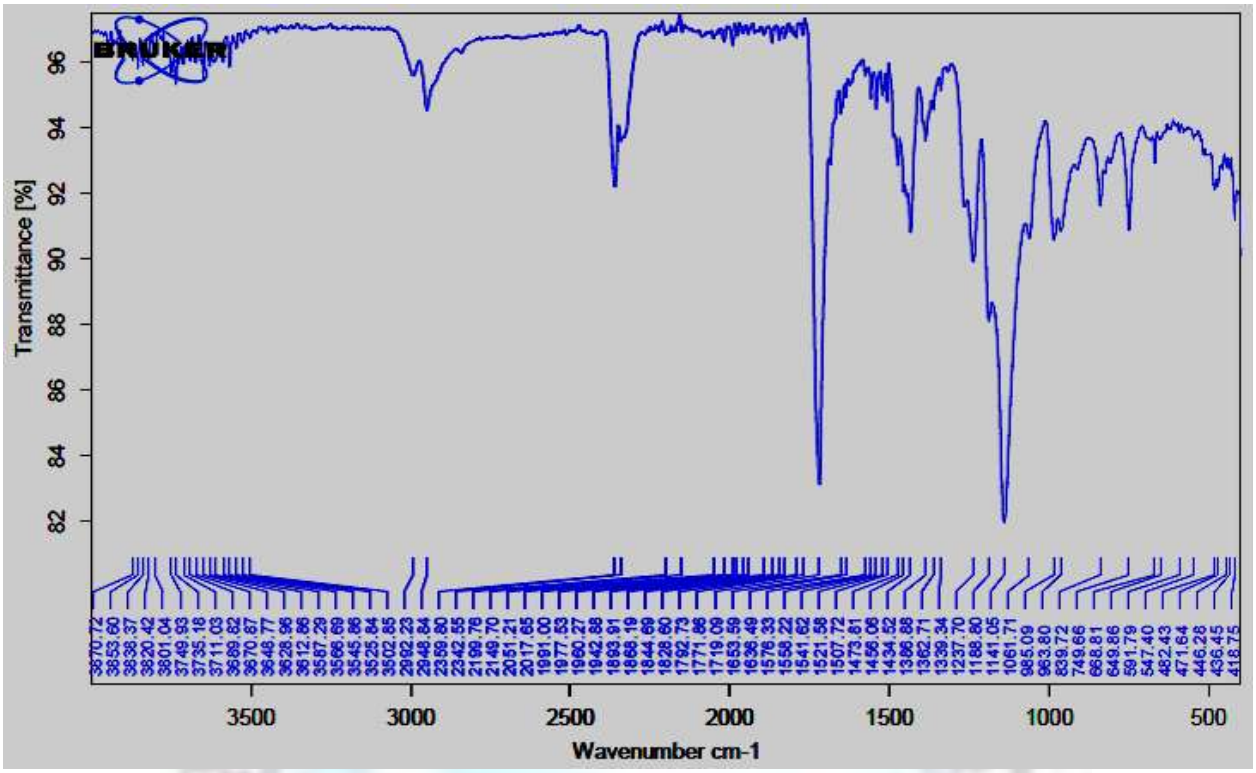

Figure (6): FTIR for 10% BMA

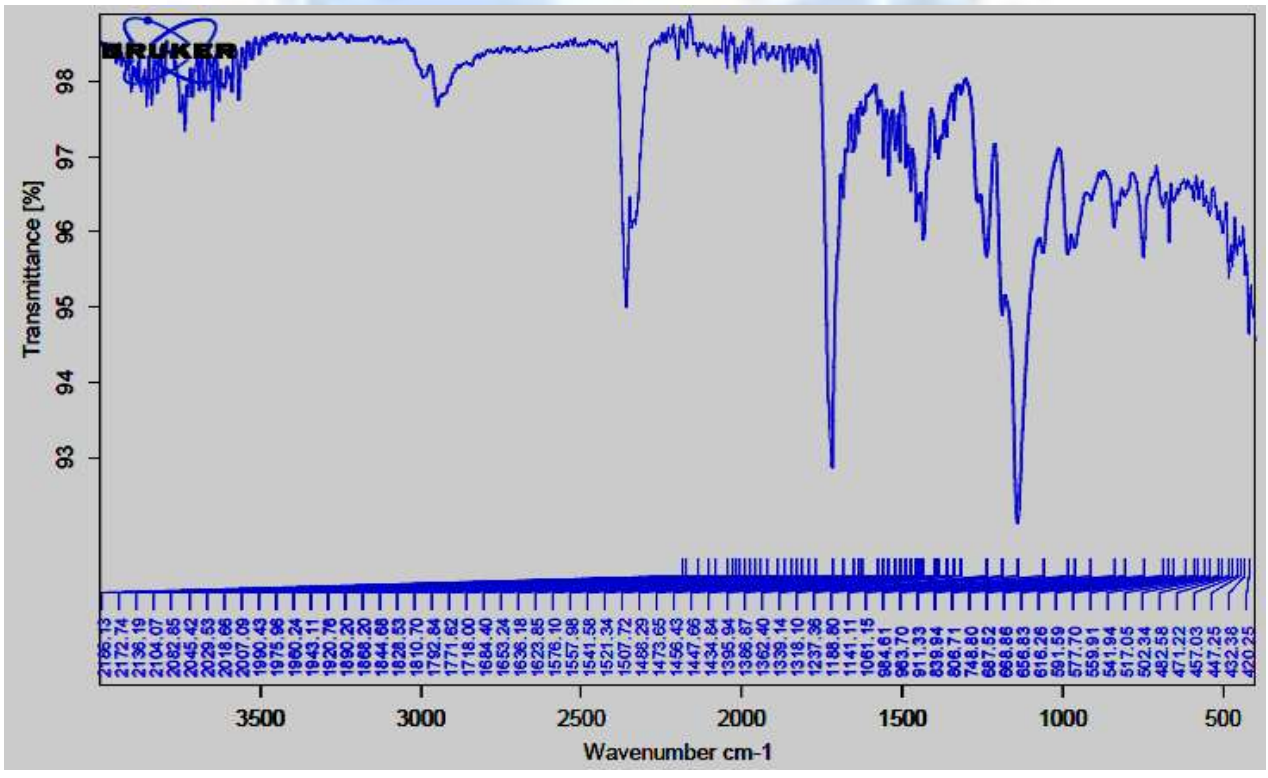

Figure (7): FTIR for 15% BMA

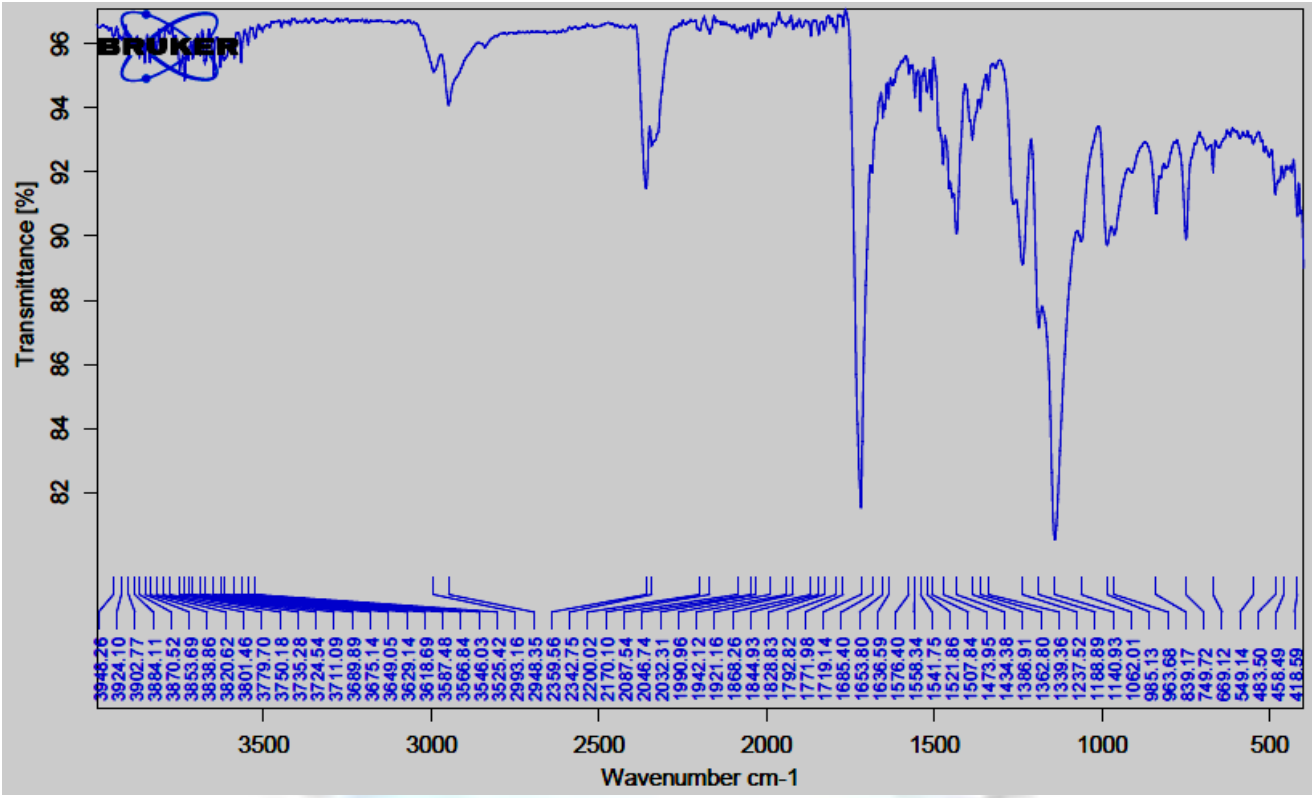

Figure (8): FTIR for 5% EA

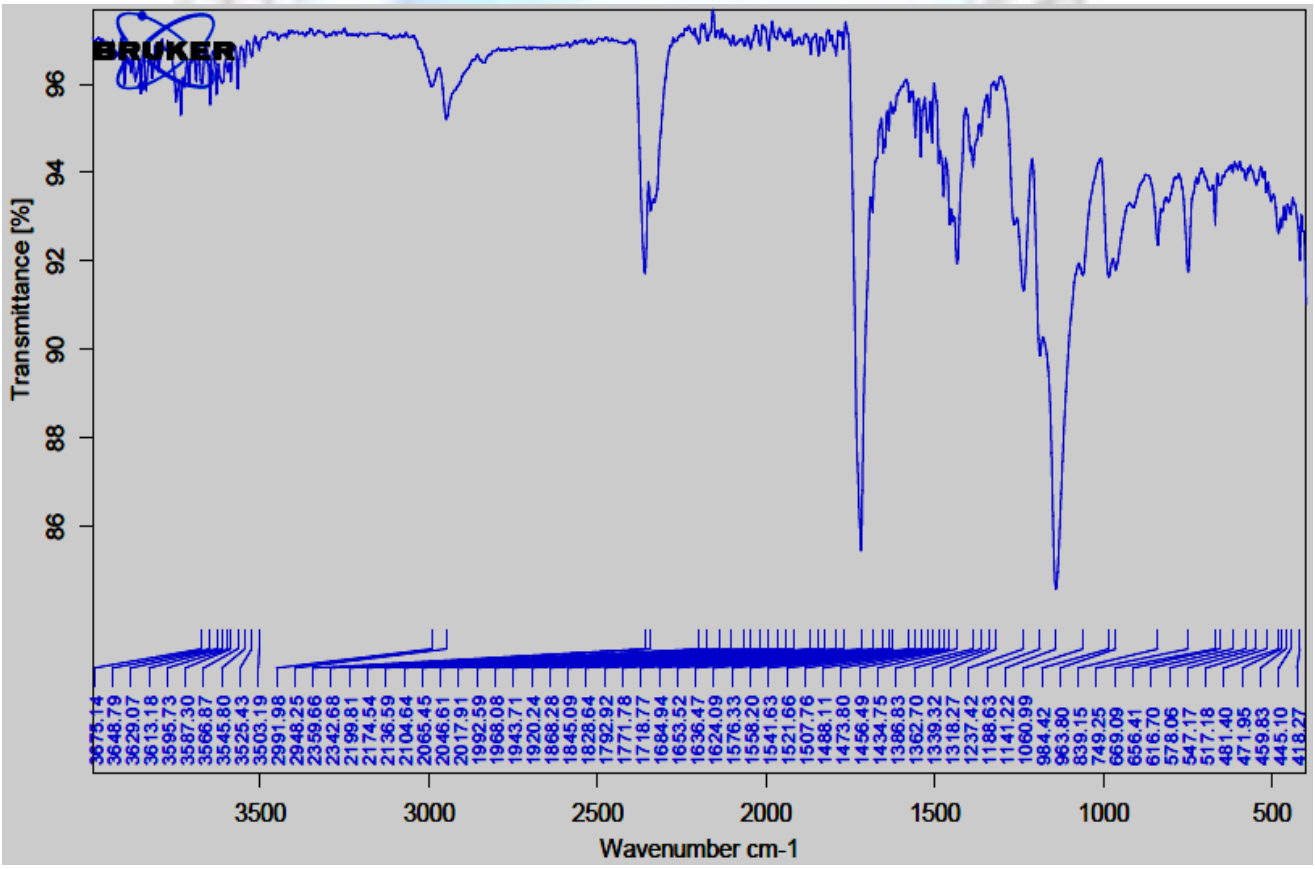

Figure (9): FTIR for 10% EA

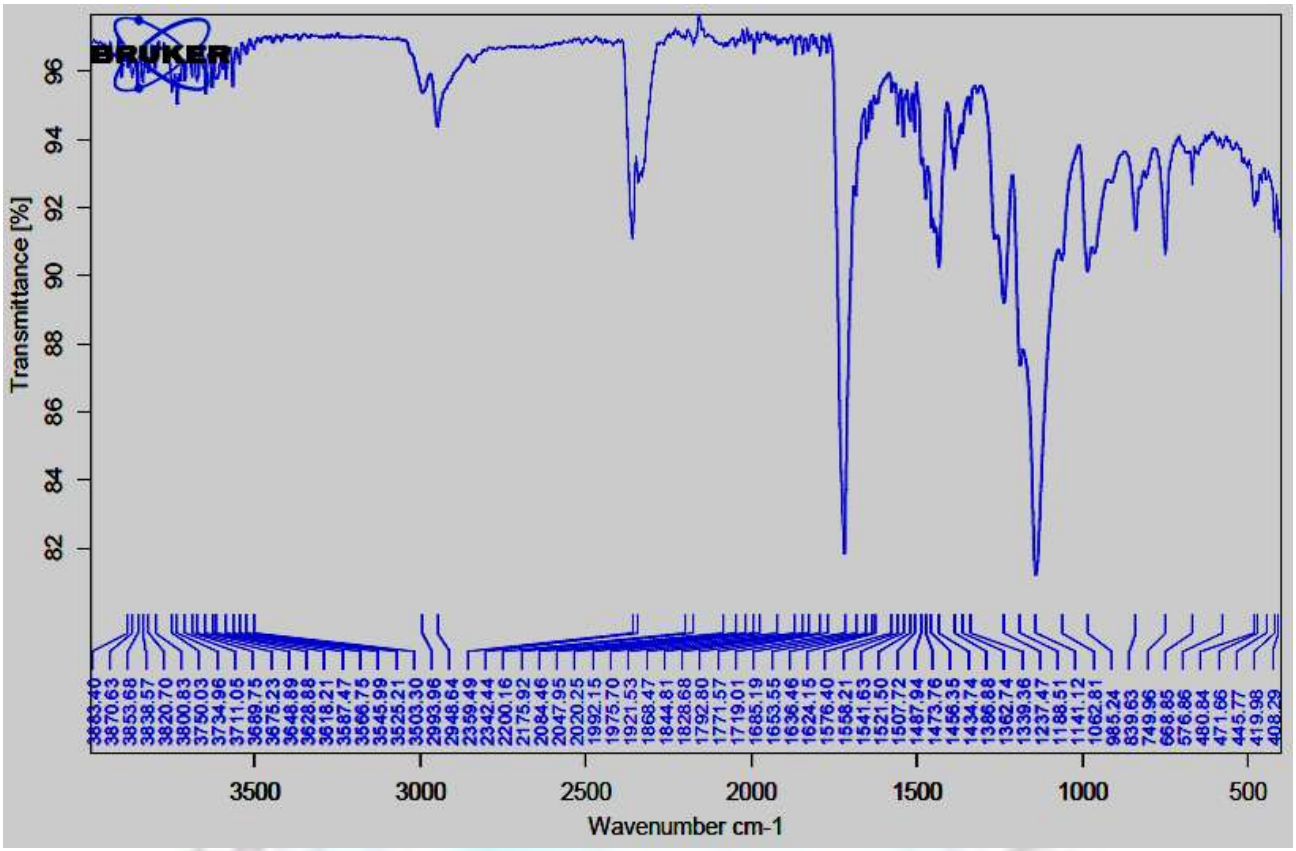

Figure (10): FTIR for 15% EA