



Investigation Of Crystallinity And Intermolecular Interaction In Ethyl Cellulose (EC) And Polymethyl Methacrylate (PMMA) Polyblend Doped With Oxalic Acid

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Abstract: Fourier transform infrared (FT-IR) and X Ray Diffraction (XRD) spectroscopy measurements for Polymethyl Methacrylate (PMMA) and Ethyl Cellulose (EC) polyblends (1:2 and 1:3) doped with different percentages (0%, 5% and 10%) of Oxalic Acid in tetrahydrofuran (THF) at room temperature were prepared using isothermal evaporation techniques. Polymers like Ethyl Cellulose (EC) and Poly Methyl Methacrylate (PMMA) being essentially insulating materials, the number of free charge carriers is very small and their mobility is very low. These measured spectra were then used to evaluate the optical energy gap in relation to blend composition. X-ray diffraction (XRD) was used to identify the molecular interaction arising in the mentioned polymer blend films. The peculiar deviation confirms the structural changes in the prepared samples.

Index Terms - EC, PMMA, Oxalic Acid, Tetrahydrofuran, FT-IR, XRD.

1. INTRODUCTION

The polymer blends are combination of different polymer-matrix composites, a material important to the electronic industry for its dielectric properties in the use of capacitors. The physical mixing or blending of two polymers produces an alloy with quite different properties, which can be potentially useful. The interest in organic and polymeric semiconductors has arisen, particularly because of their electro photo graphic and solar cell applications. Polymer blending is an attractive route for producing new polymeric materials with tailored properties without having to synthesize totally new materials. Other advantages for polymer blending are versatility and simplicity [1]. There are many investigations on polymer blending. The most important of the cellulose ethers is ethyl cellulose (EC). Deshmukh *et al* [2] reported electrical conduction in semiconducting PVC–PMMA thin film. Microhardness and X-ray diffraction studies on polymer blends of polyethylene methacrylate (PEMA) and polyethylene oxide (PEO) have been reported by Awasthi and Bajpai [3]. PMMA is widely used in many technological applications because of its unique combination of excellent optical properties with chemical inertness, some good mechanical properties, thermal stability, electrical properties, weather resistance and easy shaping were studied by Silva *et al.* [4] Ramadan *et al.* [5] added PVC to PMMA to overcome the problem of rigidity of single polymer and they observed that resultant blends were soft and open to more uses and applications. Many authors Soman and Kelkar [6], Ramesh *et al.* [7] suggested that interaction between PMMA and PVC occurred via hydrogen bonding type of specific interaction involving via hydrogen of PVC and the carbonyl group of PMMA. The changes in the values of the optical energy gaps, dielectric constants, and the refractive index under different frequencies and blending ratio were related to the effects of structural changes in the amorphous domains, impurities, and space charge existing in the interfaces between the mixed phases were studied by Belsare *et al.* [8].

3.1.2 FTIR of 1:2 EC-PMMA blend with dopant

Fig. 1(b, c) shows FTIR spectroscopy used to examine the interaction between 1:2 EC-PMMA blend with dopant.

The peak at 1192 cm^{-1} , 1184 cm^{-1} , 1184 cm^{-1} , 1151 cm^{-1} , 1054 cm^{-1} , are attributed to C–O–C stretching of EC in cases of 1:2 EC-PMMA OA (5), EC-PMMA OA (10), respectively. The peak at 1764 cm^{-1} , 1715 cm^{-1} for EC-PMMA OA (5) are assigned to C=C stretching in OA. Peak at 1267 cm^{-1} , EC-PMMA OA (5) is attributed to C–O–H bending in OA. As concentration of OA increases peak at 1764 cm^{-1} , 1715 cm^{-1} , 1267 cm^{-1} shifted to higher wavelength.

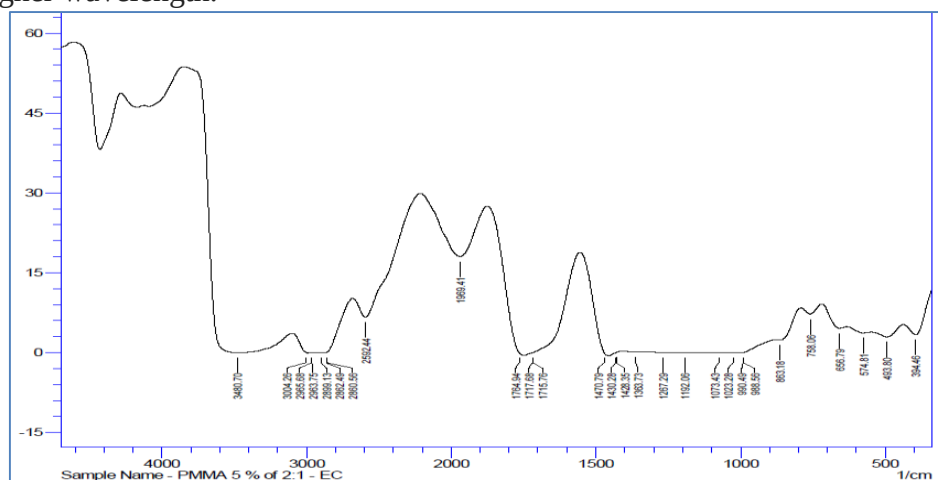


Fig.1(b) 1:2 EC-PMMA OA (5%)

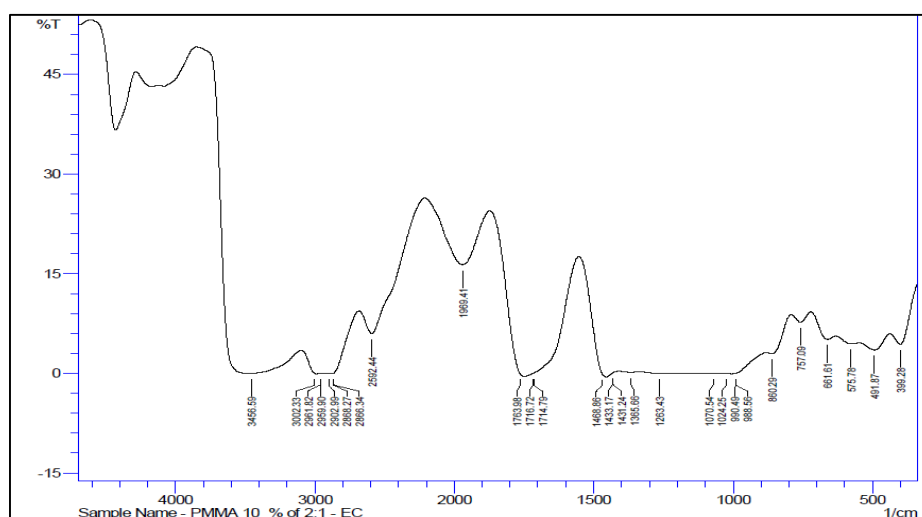


Fig.1(c) 1:2 EC-PMMA OA (10%)

3.1.3 FTIR of 1:3 EC-PMMA blend without dopant

In Fig.2, FTIR spectroscopy was used to examine the interaction between 1:3 EC-PMMA blend in which Fig.2(a) shows interaction between 1:3 EC-PMMA blend without dopant.

The absorption band at 2855 cm^{-1} corresponds to symmetric CH_3 stretching respectively is clearly seen in figure 2 (a) [9]. The peak at 1207 cm^{-1} , 1290 cm^{-1} and 1628 cm^{-1} attributed to C-O-C stretching, C-H bending and C=O carbonyl stretching respectively in EC [10]. The peak at 1770 cm^{-1} , 667 cm^{-1} , 1290 cm^{-1} attributed to C=O stretching, C-H wagging, bending of C-H, in PMMA respectively [11].

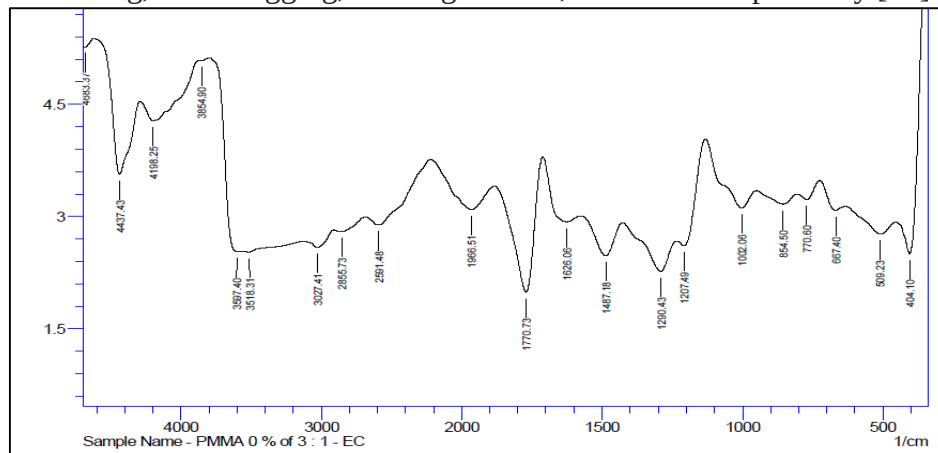


Fig.2(a) 1:3 EC-PMMA OA (0%)

3.1.4 FTIR of 1:3 EC-PMMA blend with dopant

Fig. 2(b, c) shows FTIR spectroscopy used to examine the interaction between 1:3 EC-PMMA blend with dopant.

Peak at 1770 cm^{-1} , 1768 cm^{-1} , 1762 cm^{-1} , 1789 cm^{-1} , 1760 cm^{-1} are attributed to C=O stretching of PMMA increases of EC-PMMA OA (5), EC-PMMA OA (10) respectively. The peak at 1629 cm^{-1} , 1554 cm^{-1} for 1:3 EC-PMMA OA (5) are assigned to C=C stretching in OA. As dopant concentration increases peak at 1614 cm^{-1} , 1554 cm^{-1} and 762 cm^{-1} , shifted to higher wavelength. The observed FTIR data of oxalic acid was well supported by the literature data [6].

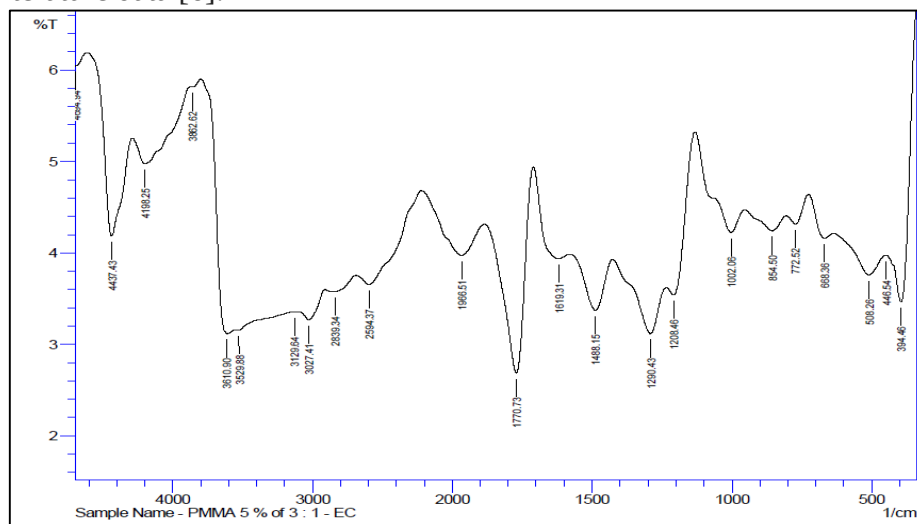


Fig.2(b) 1:2 EC-PMMA OA (5%)

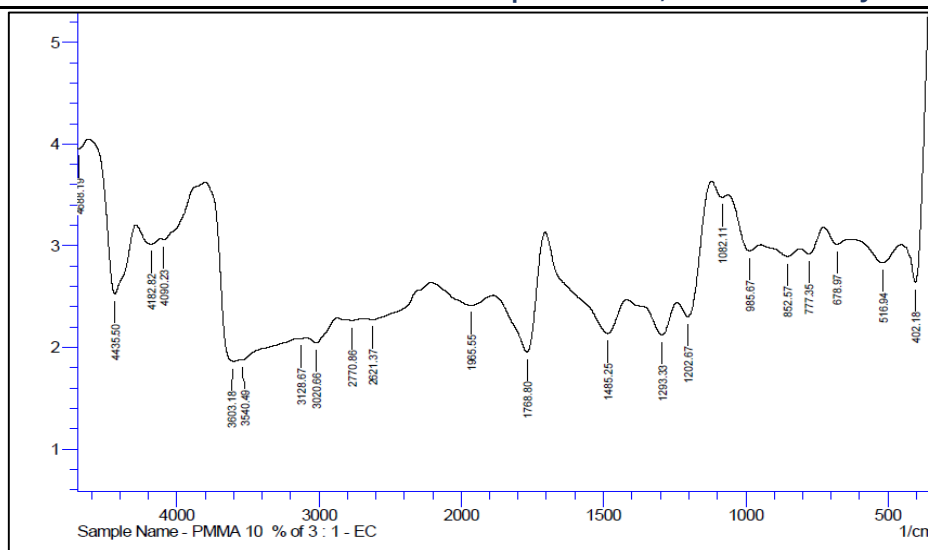


Fig.2(c) 1:2 EC-PMMA OA (10%)

3.2 XRD:

The XRD diffraction scans of the biopolymer blend are also used as a criterion to determine its overlap and homogeneity. If two biopolymers have low biocompatibility and are immiscible, then each biopolymer would have its own crystal region in the blend films.

Generally, polymer consists of both crystalline and amorphous region. Their conductivity properties are influenced by the presence of amorphous nature.

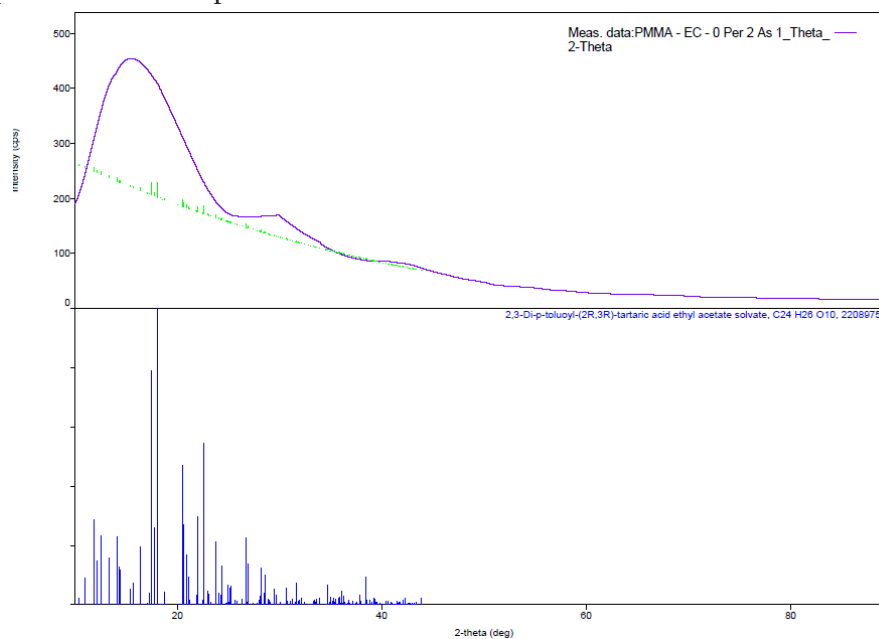
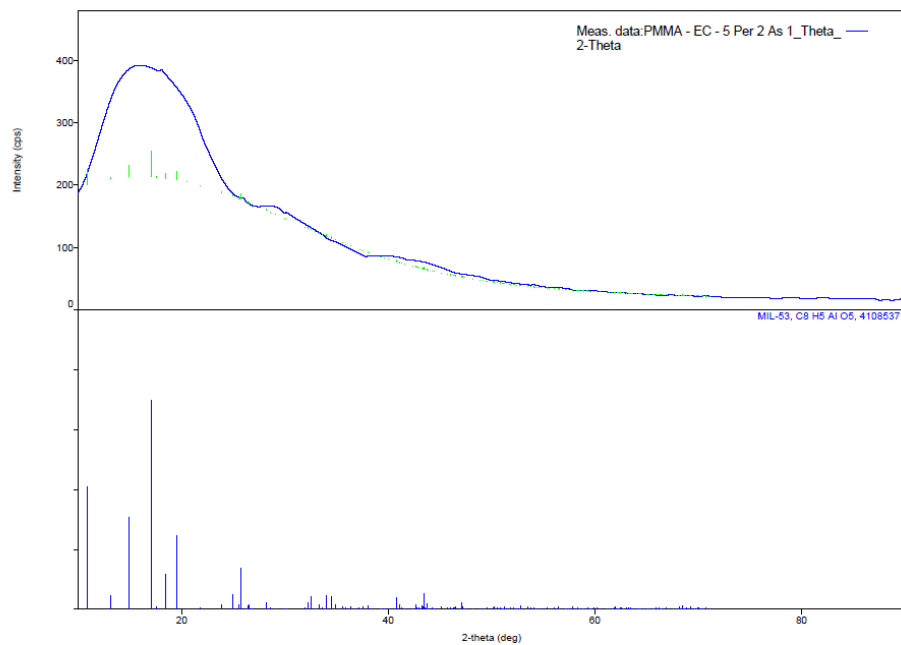
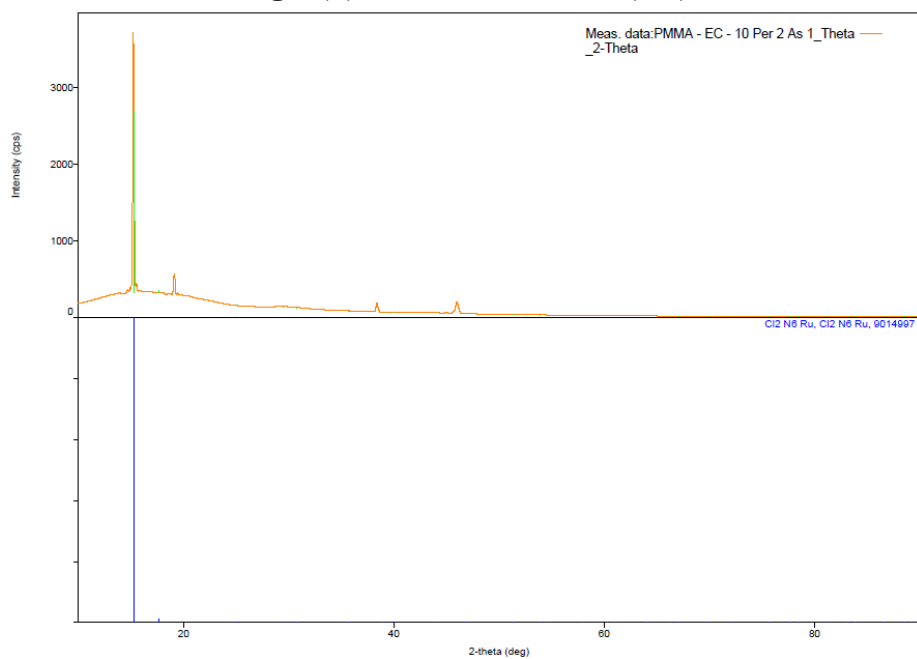
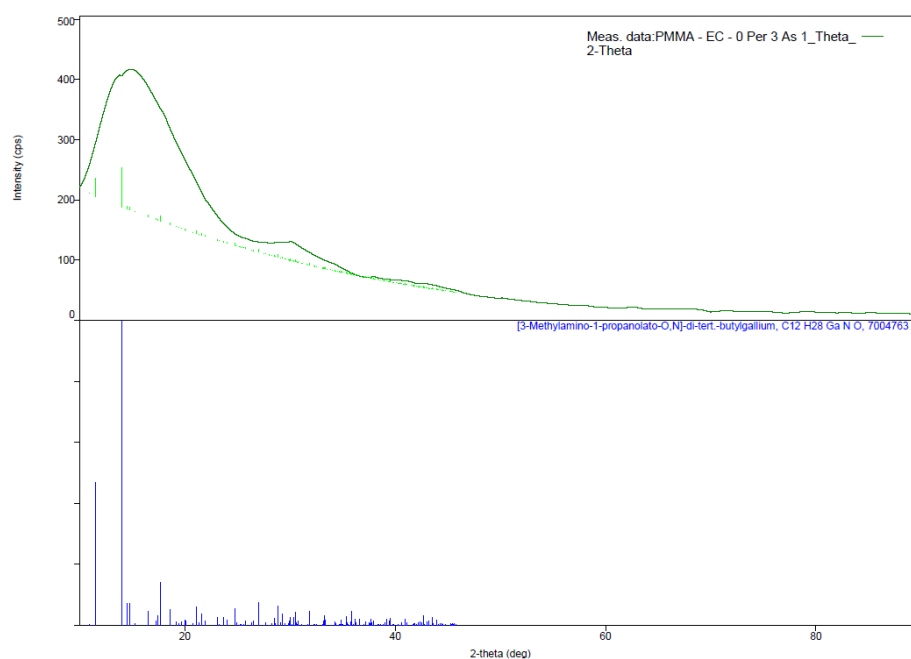


Fig. 3(a) 1:2 EC-PMMA OA (0%)

**Fig. 3(b) 1:2 EC-PMMA OA (5%)****Fig. 3(c) 1:2 EC-PMMA OA (10%)****Fig. 4(a) 1:3 EC-PMMA OA (0%)**

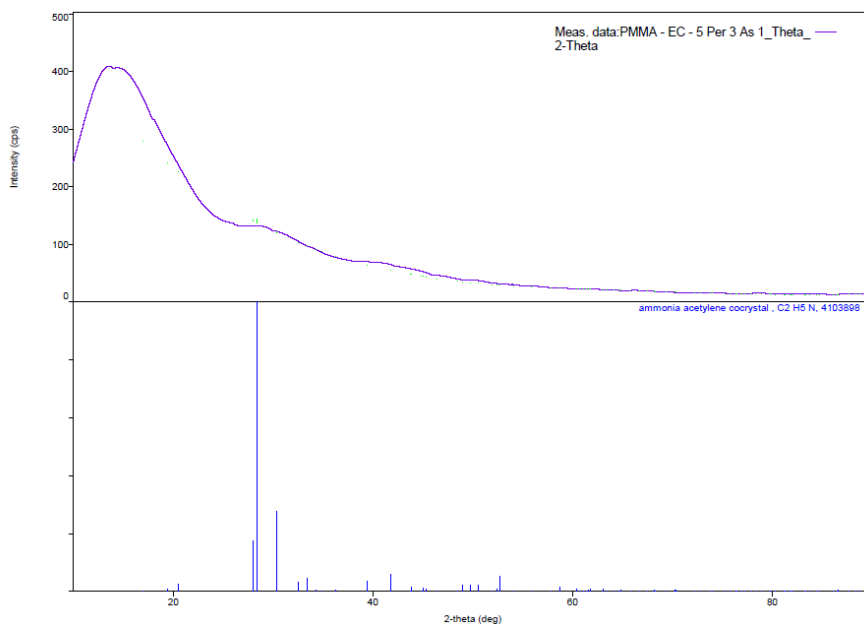


Fig. 4(b) 1:3 EC-PMMA OA (5%)

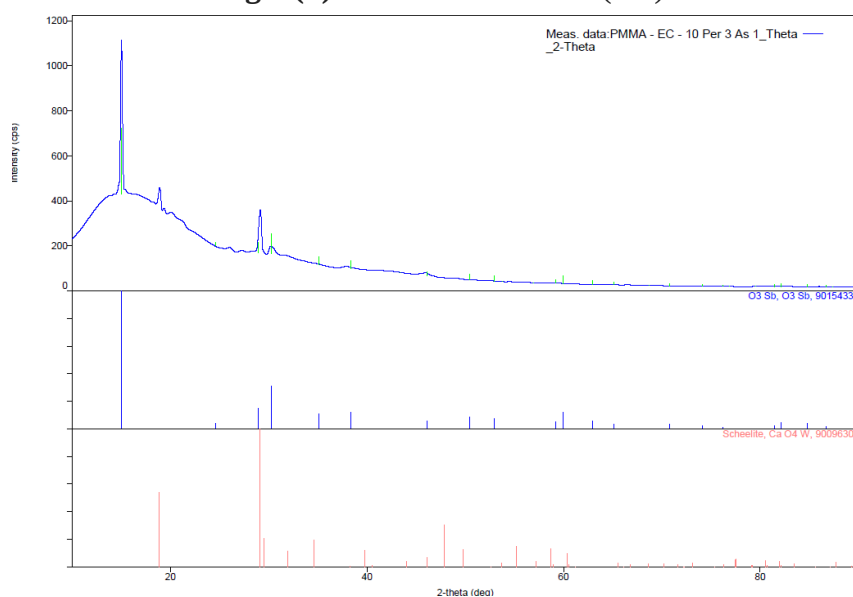


Fig. 4(c) 1:3 EC-PMMA OA (10%)

According to literature, it was observed that the broad peaks in a diffractometer were at around 18.92 degree for EC and strong broad band at 14.2 degree for PMMA [12].

From above Fig.3 (a-c) and Fig.4 (a-c), we observe that the sample shows noisy spectra. It is obvious that the X-ray pattern of synthesized PMMA shows a strong broad band at $2\theta = 14.2^\circ$ and two peaks at $2\theta = 30.2^\circ$, 2θ and 42.3° shows the good agreement with the JCPDS card no. 13-0835 [13]. The observed scans of pure EC and PMMA blend as shown in Fig. 3(a) and Fig. 4(a) exhibits a characteristic broad amorphous hump observed around $2\theta = 14^\circ$ to 18° and this means that EC well mixed with PMMA which indicated that EC/PMMA blend is amorphous [12, 13].

Some new peaks were observed in XRD patterns by doping with oxalic acid with different ratio only one sharp peak is obtained at 14 to 18 degree which is shown in Fig 3(c) and Fig. 4(c). Broad peak of pure EC and PMMA blend is decreased due to the doping of oxalic acid in main chain of EC and PMMA blend [12]. This study reveals that 2θ and d-spacing values of doped samples are different from that of pure EC/PMMA blend which confirms the presence of oxalic acid in the samples. Due to the slight changes in intensity of some peaks were observed as a result of doping. There is also a slight change in the lattice parameters of the doped crystals. The slight change in the intensity of peaks and lattice parameters may be due to lattice distortion by doping in the parent crystal. Hence it is revealed that dopant can enter in the lattice of oxalic acid without causing much distortion [14].

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