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Influence of non-thermal plasma treated Nano-carbon on TiO_2 coated PMMA for bio activation of medical devices

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Abstract: The Titanium di Oxide (TiO_2) was successfully coated over transparent Polymethylmethacrylate (PMMA) sheets by spin coating method which was then subjected to Acetylene Plasma processing to form a thin- Nano carbon layer on the TiO_2 coated PMMA. The amorphous carbon was uniquely coated by novel vacuum plasma based immersion technique on TiO_2 coated PMMA with the base pressure of $5x10^{-5}$ m.bar. The Acetylene gas was used as Plasma gas which was the ultimate source of carbon. The afore said incorporation technique helps PMMA to attain more mechanical strength, improved surface and mainly good bioactive property with high antibacterial activity with the continuous carboxylic group of polymer network. This plasma processed TiO_2 coated PMMA was subjected to various characterization techniques such as Raman spectroscopy, FTIR and XRD analysis. The carboxylic stretching mode and C=O bending modes were evident by Raman spectroscopy studies were as the symmetric and asymmetric carboxyl groups were observed by FTIR spectroscopy. Three humps were observed as an amorphous Nano carbon at 10 and 45 2θ values using XRD measurements. This study confirmed the formation of thin- Nano carbon layer on the TiO_2 coated PMMA which may be used for medical device applications such as for bio-implant purposes after a detailed investigation.

Keywords: PMMA, Non-thermal Plasma processing, Raman Spectroscopy, Spin coating

1. Introduction

The polymers are now one of the most essential materials of the world that has importance in usage of our day to day lives. In polymers large number of materials, made up of small molecules and monomers are linked to form the long chain and are use of the much more products in our daily usage. Polymers have been around us in the characteristic world since the absolute form starting point (for example Cellulose, starch, and normal elastic) to the man-made polymeric materials to have been concentrated since the centre of the nineteenth century. Today, the polymer business has quickly evolved and is bigger than the copper, steel, aluminium and some different ventures combined. It is hard to envision human culture without manufactured and common polymers [1]. The bio-active polymeric materials are one such class of polymers that are used to the orthopaedics and traumatology. The metals, ceramics and polymers have a coefficient of elasticity on the cortical bone and can successfully be utilized in the suppression of the strain shielding between the implant and the bone. The polymers offer easy manufacturing production of various shapes (films, fibres, sheets, special implant shapes, etc.) that afford a base to use the polymeric material as high-quality biomaterials. Polymeric bio-materials need to be biocompatible, sterilizable; they require adequate mechanical, physical and surface properties (e.g., required roughness, adhesive properties, etc.).

Bio applications for implants generally are applied in direct contact with the human living body and blood. The surface and biological properties assume a vital job concerning the biomedical polymers for medical applications. In any case, the bio-polymers can be utilized for medical applications, which are a mind boggling and convoluted subject.

The surface and biological properties are of high significance; however, the safeguarding of the necessary mechanical properties is also required simultaneously. Numerous conceivable treatment techniques exist to arrive at this objective. The vast majority of them depend on synthetic systems, which can be unsafe. The plasma surface treatment is one of such techniques that offer free change prospects to the surface and biological properties with no adjustment or alteration to the mass (mechanical) properties [2].

Mitsugi *et al.,* (2008) have developed a new type of bead-mill assisted PMMA/TiO_2 composite by using subsequent polymerization method and the average particle size about 10 nm. With the addition of silane the bead-mill can able to break the agglomerated particles where the mass fraction of particles has high as 0.05. The prepared Nano composite improved the UV absorbing and high thermal stability. Their newly developed bead mill is easy operating and simple method to make nanoparticles into organic solvents to perform very good option to produce Nano composite synthesis [3].

Ksapabutr *et al.,* (2008) coated titanium dioxide crystal over the PMMA sheets by using spray pyrolysis technique under high electric field. Processing condition, precursor concentration and calcination temperature for the morphology of the particle was taken into consideration. The main usage of the composite is to develop the light radiation was observed by LUX meter.

PMMA/TiO_2 composite gave high intensity of light scattering than PMMA sheet. As the TiO_2 concentration increased the brightness increased for which the calcination temperature was about 800°C at the rate of concentration of 2.4 mgl [4]. W.Su et al., (2010) modified the PMMA surface by using TiO_2 nanoparticles to improve the bacterial activity. TiO_2 films was coated on the Plasma treated PMMA by using Sol-Gel dip coating technique. This surface modified PMMA/TiO_2 was studied for several characterizations and for bactericidal property with Staphylococcus aureus (gram positive) and Escherichia coli (gram negative). Anatase structure of TiO_2 was present with the average particle size of 4 nm. The surface was of hydrophilic property and improved roughness. As prepared TiO_2/PMMA obtained light induced and anti-adhesive bactericidal property with photo catalytic inactivation [5].

Amit Chatterjee *et al.,* (2010) have discussed about the materials property improvement of PMMA using TiO_2 nanoparticles. Preparation of TiO_2/PMMA was done by using twin screw extruder method to improve the polymers performance. The mechanical property drastically was found to increase up to 90% and UV absorption got improved up to 4 times. Sample performance depended on the dispersion of the TiO_2 into PMMA. The physical and chemical properties were found improved because of addition of TiO_2 in the point of degradation among the PMMA concentration [6].

Sadiqali cheruthazhekatt *et al.,* (2010) had studied the artificial bio-materials for the functionalization of plasma treated surface to increase the rate of drug loading and controlled release for long term. In the tissue improvement; the plasma played the vital role of improving the bioactivity of the scaffold and promoted cell adhesion. Compared to the other treatment methods the plasma technique created an unique surface property and ascertained the convention of plasma-medicine as a fast development of the medical industry [7].

Julius Andrew p.nunez *et al.,* (2017) discussed the antibacterial efficiency of the TiO_2 coated PMMA sample against the E.coil bacteria with a reduction of bacteria 70% to 80%. They further discussed the photo-induced hydrophilicity property which illustrated the contact angles for the TiO_2 coated PMMA was less than the angle of the pure PMMA. The decrease of the contact angle of a surface was observed with an increase in the surface roughness property. The presence of TiO_2 on the PMMA sample was confirmed by the XPS, FTIR and EDX analyses [8].

Hiroki Nagasawa *et al.,* (2018) developed an Atmospheric pressure plasma enhanced chemical vapour deposition method to form TiO_2 on polymers to improve the UV stability. The TiO_2 had amorphous structure over the PMMA without disturbing the polymers structure. The composite material of PECVD processed TiO_2 absorbed the UV light around 330 nm. TiO_2 coated PMMA presented more amount of UV absorption around 99% at the range of 200-280 nm. The PECVD based TiO_2 coated PMMA sample was found to be a very good UV shield to prevent UV degradable polymers [9].

Ghaith darwish *et al.,* (2019) in their paper had discussed, modifying the surface properties of PMMA polymer by using the atomic layer deposition technique (ALD) to deposit TiO_2 -Nano film in PMMA at the 65°C.They also successfully decreased the water contact angle of after TiO_2 coated PMMA less than from 70° to 5°.The main advantage of TiO_2 coating improved the wettability, wear-resistance and the resistance for the microbial adherence. The major application of this work was to overcome the limitations and improvement of mechanical strength [10].

2. Experimental procedure

2.1 Poly (methyl methaacrylate) PMMA

Poly (methyl methaacrylate) PMMA, having the IUPAC name of poly (1-(methoxy car-bonyl)-1-methylethylene) to the hydrocarbon standpoint, and the poly (methyl 2 –methyl-propenoate) from the ester standpoint, to the synthetic polymer from the methyl methacrylate monomer. PMMA was introduced in the mid of 1930s by the British chemists, R.Hill. PMMA is an optically clear (transparent thermoplastic, and it is generally utilized as a substitute for inorganic glass, since it shows high effect quality, is lightweight, shatter-resistant, and displays great handling conditions remarkable properties incorporate climate obstruction and scratch opposition. The presence of the adjacent methyl group (CH_3) in the polymer structure keeps it from pressing intently in a crystalline manner, and from pivot around C-C bonds. This is the reason PMMA was seen as an undefined thermoplastic. The major application of polymers used in the world war time, when the PMMA utilized as air-craft windows and gun turrets [11].

PMMA is one of the most advantageous polymers for the optical applications, it offers good optical properties, high moulding precision and low expenses, and a covering of the delicate material is fundamentally planned to improve its mechanical solidness. Additionally, antireflection covering is fundamental for some optical applications [12].

PMMA also called as the acrylic or Plexiglass and the poly methyl methacrylate has been applied to the anti-corrosive coatings, optical filaments, focal points, bone concrete, contact and intraocular focal lens, dental resins and prostheses and others. It is the rigid, transparent, biocompatible and low cost polymer with high resistance [13].

2.2 Physical properties of PMMA

In the *Acrylite* family one of the amorphous polymers is PMMA. The major property of the PMMA was clear and colourless polymer. The glass transition temperature range of the PMMA is 100°C to 130°C. The density of the PMMA is 1.20g/cm^3. PMMA melts at 130°C and its water absorptivity is 0.3% and the linear shrinkage mould of PMMA is 0.003 to 0.0065 cm/cm. The PMMA withstand temperature is high as 100°C and low as -70°C and the good degree of compatibility with the human tissue and the refractive index of 1.49. The mechanical characterization of the high young's modulus and a low elongation to the breakage [11]. Samples of PMMA films are shown in Figure 1.

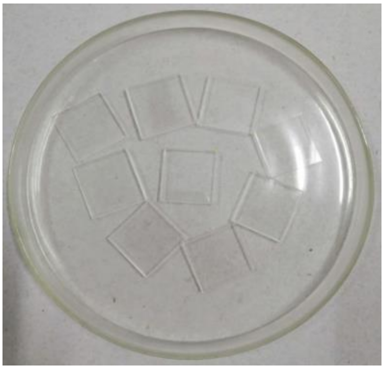

Figure 1. PMMA sheet

2.3 Titanium Dioxide (TiO_2)

Titanium dioxide (TiO_2) is a chemical that has much attention to its better bioactivity with anti-bacterial effect and its photo catalytic activity. The major application of TiO_2 comprises the self-cleaning function, good oxidizing stability and the antibacterial activity. TiO_2 compared to the other products is good as to be used in the self-cleaning products and they have non-toxicity and environmental goodness. TiO_2 applied as coatings to the surface of the materials can be used for a variety of applications. The coating of the TiO_2 on PMMA can be used as a self-cleaning polymeric device. This would serve to empower existing clinical polymer makers to incorporate oneself cleaning idea of TiO_2 coatings into existing procedures [14].

The antibacterial effect of TiO_2 was reported on the 1985, Matsunaga also it is good for treatment of waste water [8].

2.3 Spin Coating Technique

Spin coating is the simplest method for fabricating film on a substrate. The spin coating process starts with the dilution of the material to be deposited through a solvent. The resultant is subsequently dispensed on the substrate surface. The wafer is then spun at a high speed. The thickness of the film is decided by the spinning speed, physical phenomenon, and viscosity [15]. The *sol* of the material to be coated is deposited onto the substrate which is spun off at a high velocity during a range of 1000-8000rpm and leaving a consistent layer [16]. The spin-coating processes are often divided into three stages. Deposition and spin up, spin off, and film drying. Although those stages overlap slightly, their physics are often effectively modelled by separating them into three distinct stages [17].

Deposition and spin up: within the first stage and more than liquid is delivered to the substrate at rest or spinning slowly [18]. The liquid solution could also be deposited in several different manners starting from pouring the whole solution at the middle of the disk and allowing it to hurry radically over the film, to delivering the liquid during a steady stream moving radically over the rotating disk in order that the deposited liquid forms a spiral [17]. The spin up stage full speed of several thousand revolutions per minute. Most of the gas originally in touch with the substrate surface is displaced because the dynamic wetting line at the front of the liquid advances radically outward [18].

Spin off: The key to spin coating is that the bear stage [18]. During this stage the film is thinned thanks to a mixture of convection and solvent evaporation. The centrifugal forces act to drive the fluid radically off the sting of the disk impeded only by the viscous resistance. This radial flow quickly diminishes because the film has become exceedingly by several orders of magnitude. During fluid flow the film is additionally thinned by solvent evaporation to the over-lying atmosphere. It is the trade-off between those two mechanisms that controls the skinny film thickness, uniformity, and therefore the success of the spin coating process [17].

Film drying: during this end of spin coating, fluid flow has essentially halted and further shrinkage of the film arises from solvent loss alone. At is point bear stage end and the film drying stage begins [17].

2.4 Plasma processing

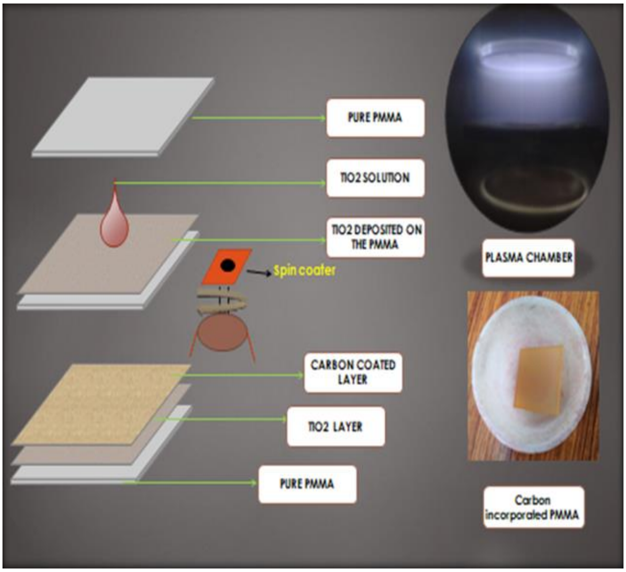

Figure 2. Schematic representation of Plasma Immersion reactor

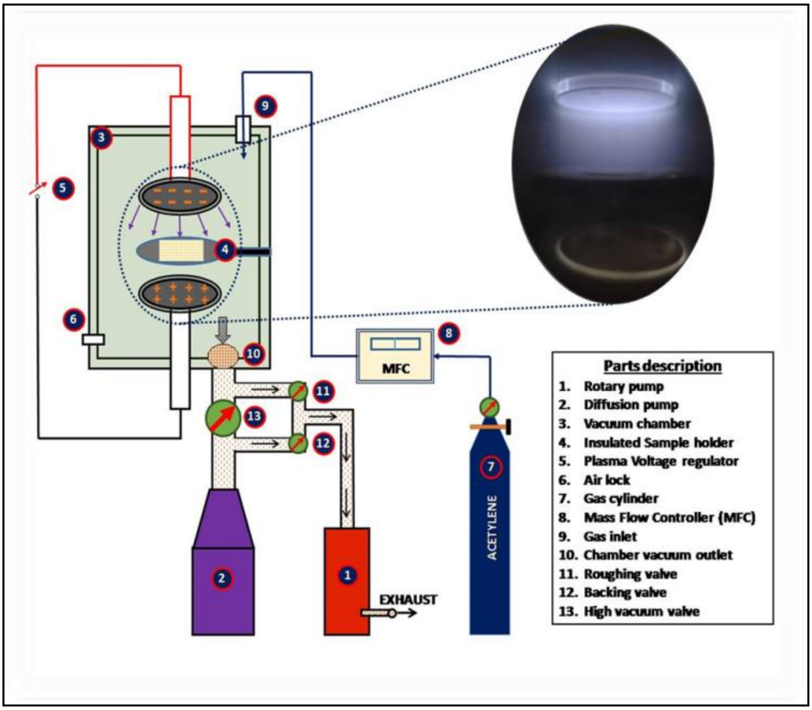

Figure 3. Schematic diagram of formation of Carbon- TiO2 coated PMMA sample.

In our work, we have mainly focused on the non-thermal DC plasma immersion technique applied to process the TiO_2 coated PMMA samples though the acetylene and argon gasses. The plasma process for the polymeric materials focused on the surface modification and for the biological applications. The plasma is produced by gaseous discharge at a very low pressure under the high vacuum conditions [19-20]. A schematic diagram of the system is demonstrated in Figure 2 and the process is explained in figure 3.

3. Result and Discussion

3.1 X-Ray Diffraction analysis

To determine the phase analysis and orientation of a single crystal or grains our instrument is for grazing incidence x-ray diffraction (GIXRD) for the thin films find the thickness of the thin crystalline films (20-200 nm depend on mean atomic number) on polished, flat surfaces The peculiar spectra of amorphous carbon which was coated on the TiO_2 coated PMMA is shown in the figure 6. The peaks were observed with corresponding to carbon and TiO_2 at 2θ values of 15° and 30°. The phase transition of PMMA and our result may be the addition of carbon which is present at 2θ value of 45°. The present nature of the sample is quite a dissociated one of the polymer Nano composite due to annealing. The diffusive scattering of

PMMA and TiO_2 composite becomes amorphous as shown in the figure 4. This diffraction spectrum alone indicated the presence of PMMA/TiO_2 composite [5, 21].

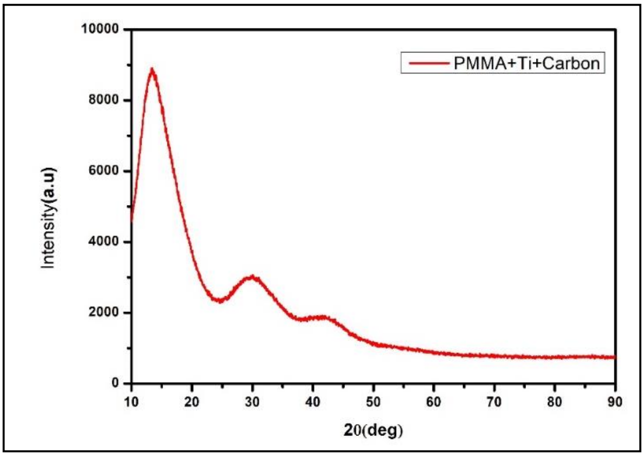

Figure 4. XRD spectrum of Titanium and Carbon coated PMMA

3.2 Raman Spectroscopic analysis

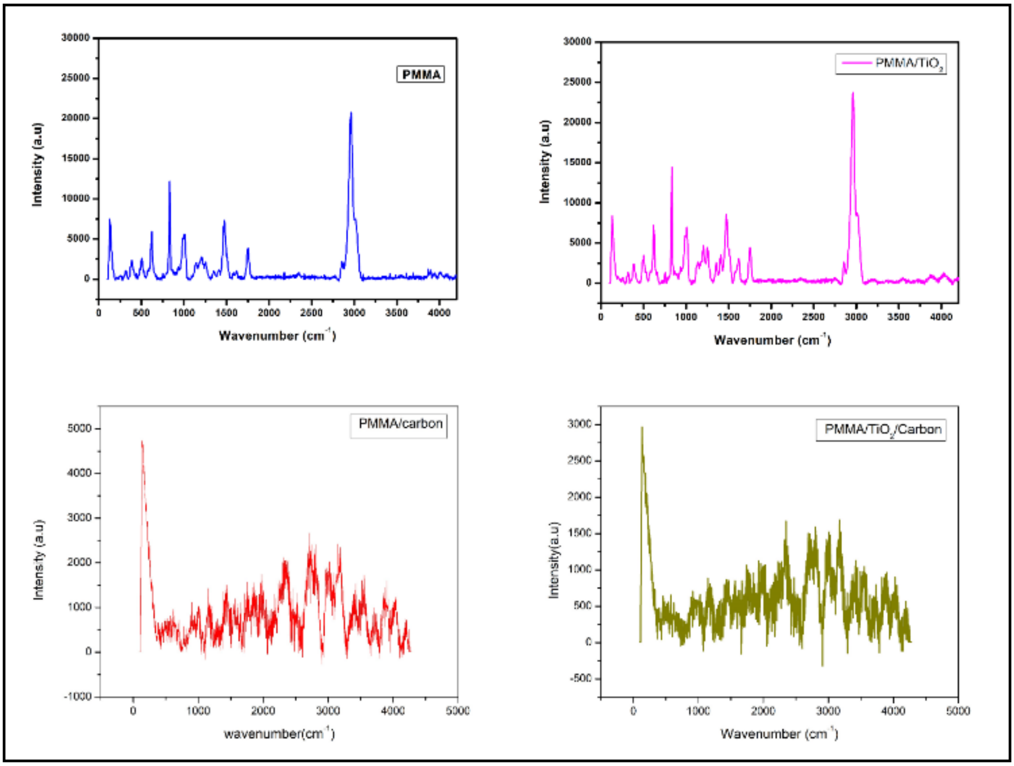

Figure 5. Raman spectra of Titanium and Carbon coated PMMA

The structural differences between the TiO_2 and Carbon over the PMMA sheet were acknowledged by Raman spectroscopy. In the visible region, Raman wavelength of π bond state is much higher reactive than σ state of carbon-based materials. Raman analysis have achieved using 532 nm green laser source is shown in figure 5. The band at 2955 cm^{-1} was observed to be the most prominent peak due to the presence of C-H stretching vibration. Bending mode of C-O-C was present at 600 cm^{-1} cordially the 850 cm^{-1} and 920 cm^{-1} remains CH_2 were assigned. Moreover, observed bands at 995, 1080, 1265, 1450, 1655 and 2900 cm^{-1} were affirmed due to O-CH_3, C-C skeletal, C-COO, C-H, both of C=C with C-COO and C-H of α-CH_3 coordinates respectively.

The variation in the frequency was observed with the Raman band by the inelastic scattering of incident radiation. Where the carbon coated over the TiO_2- PMMA was suppressed the peak intensity due to the plasma process deals higher energy particle occupying surface of the sample. A slight noise was figured out due to the amorphous structure of carbon. The phonon scattering peak at 150 cm^{-1} due to the titanium over the PMMA [22-24].

3.3 Fourier Transform Infra-Red Spectroscopy

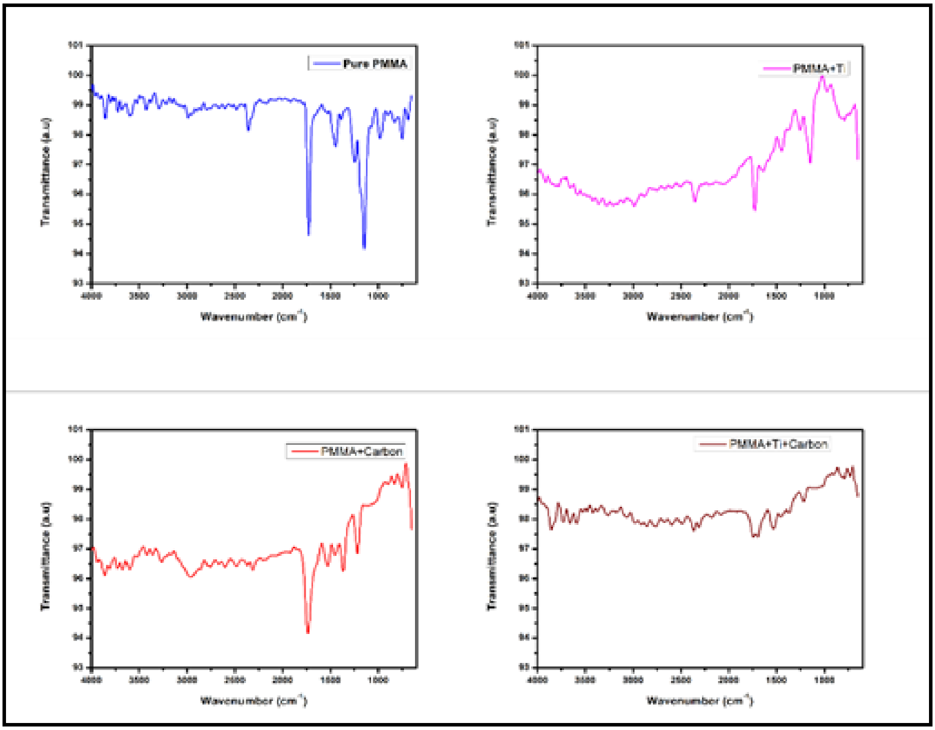

Figure 6. FTIR spectra of Titanium and Carbon coated PMMA

Fourier Transform Infrared spectroscopy (FTIR) was investigated to observe the chemical interaction of Titanium and carbon over the PMMA. The FTIR results are shown in the figure 6. Among all the spectra, the peak at 1720 cm^{-1} deals the carbonyl group. Presence and disappearance of new peaks were possibly due to the chemical interaction of TiO_2 and PMMA nano composite where the carbon impacts on PMMA lesser than the TiO_2 does. The bands give you an idea on that between the 400 to 700 cm^{-1} was present due to the Ti-O-Ti structure. Usually, the peaks of PMMA falls lower lateral due to the addition of nanoparticles. Band at 2992 cm^{-1} was present because of $-CH_2$ stretching mode and it presents all the spectra (1ftir). Asymmetric deformation vibration of PMMA/TiO_2 was present at 1480 cm^{-1} and 1720 cm^{-1} which was due to the C=O stretching of PMMA and Titanium reduces this with carbon particles even at the lesser quantity. This is the reason of the energy that engages in reaction rate of the surface which is more coercive than the pure PMMA [3, 5, 6].

4. Conclusion

The plasma processed Nano carbon, on the surface of spin coated TiO_2 on PMMA was achieved. The plain transparent PMMA was subjected to spin coating of Titanium Oxide composite to form TiO_2 coated PMMA. Then a thin Nano carbon layer was formed over the TiO_2 coated PMMA surface through plasma immersion method using the acetylene gas plasma as a predominant carbon source. The FTIR studies of the Nano carbon TiO_2 coated PMMA samples demonstrated that the carboxylic groups were found to be dominated in the samples. The amorphous carbon was observed through Raman spectroscopy with the attribution of stretching and vibrating modes existed with the Nano carbon TiO_2 coated PMMA samples. From the XRD; the amorphous peaks of PMMA/Titanium surface with the carbon at 2θ values of 15°, 30° and 45° was found. The tensile strength of the PMMA and titanium carbon composite has been known the strength of the film turn out to be increased; inappropriately, the data was not included. Finally, this work is much more different with the several aforementioned literature surveys, that the incorporated plasma process for carbon generation is a unique route attained. The biological testing of the samples is currently under progress to find the effectiveness for usage of the product in possible medical application.

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