SUMMARY
CHAPTER 5

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This work deals with the synthesis and characterisation of twenty citric acid based copolyesters. All the copolyesters have been synthesised by catalyst free melt polycondensation of diols and dicarboxylic acids with citric acid as a common monomer. Also, nano hydroxy apatite (n-HAp) by sol-gel method and the corresponding six n-HAp/polyester composites are prepared and characterised.

The structure of the repeating units of the synthesised copolyesters have been determined by IR, $^1$H NMR and $^{13}$C NMR spectral methods. IR spectra of all the random copolyesters show characteristic absorption due to ester carbonyl stretching vibrations. IR spectra of n-HAp/polyester composite shows the molecular interaction between phosphate in n-HAp and the polymer. $^1$H NMR spectra of the copolyesters exhibit signal characteristic of methylene protons of diols and diacids used. $^{13}$C NMR spectra of copolyesters have shown the characteristic resonance lines due to carbons present in different environments.

The synthesised copolyesters are soluble in common solvents such as acetone and chloroform which helps in the easy processing of the polymers. The molecular weights of the two synthesised polymers have been determined using MALDI-MASS spectral analysis.

The thermal analysis of the synthesised polyester and the composites have been carried out by Differential Thermal Analysis, Thermogravimetric Analysis and
Differential Scanning Calorimetry. Thermal properties such as glass transition temperature, melting temperature and decomposition temperature are measured. The thermal stability of the synthesised copolyesters increases with increase in methylene units in the polymer chain. Thermograms shows the glass transition temperature values polyesters are below room temperature which is a characteristic feature of elastomeric behaviour.

The mechanical properties of the thin film of polyester and the n-HAp/polyester composites are studied. Tensile strength, Young’s modulus and elongation at break are measured and compared. The mechanical properties of the synthesised polymeric nanocomposites are comparable to those of natural extracellular matrix components which are soft and elastic polymer networks providing mechanical stability to tissues and organs.

X-ray diffractograms of the nanohydroxyapatite (n-HAp) prepared at different temperatures have been recorded and the crystallite size are calculated using Scherer formula. Also, the X-ray diffractogram of the polyester and the corresponding n-HAp/polyester have been recorded and compared.

Scanning Electron Microscopy analysis of the nano-hydroxy apatite, polyester and the composite are carried out and morphologies are examined. The image shows the agglomeration of nanosized grains of hydroxy apatite. The structure and morphology of nanohydroxy apatite has been confirmed by Transmission Electron Microscopy which shows the spherical shape morphology in the nanometer scale of the dimension of 30 to 70nm.
Cytotoxic activity test of n-HAp/Polyester composite has been carried out which indicates that the prepared nanocomposites shows better biocompatibility over cell growth. Porous scaffold of the n-HAp/Polyester is fabricated by particulate-leaching technique and SEM images are taken. SEM images show the appearance of pentagonal pore shape in the porous scaffold which is useful in tissue engineering applications.

Upto the recent past, researchers working on nanocomposites which are useful in limited fields but more research is necessary for polymeric nanocomposites in order to meet the growing demands in the medical field. This is possible by synthesising novel polymeric nanocomposites with good mechanical, thermal and biocompatible properties with potential medical applications. In this point of view, the present investigation has been started by our team. It is being continued to achieve the desired goal.