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**Preparation and characterization of bioactive polymeric
hydrogel based on starch for bone regeneration**

**A thesis submitted in partial fulfillment of the requirements for the degree of Master
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By

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SUMMARY

In the biomedical field, hydrogel systems have gained a lot of attention owing to their excellent physical, chemical, mechanical and biological properties. Currently, many natural and synthetic polymers are being widely explored for the fabrication of hydrogels which have been studied and used in regeneration of cartilage or bone, owing to their biodegradability, biocompatibility, nontoxicity, and availability.

This work was focused on the preparation and characterization of biocompatible, low cost, and efficient new injectable bioactive composite hydrogels by microwave assisted grafting in heterogeneous aqueous solution, and use it for regeneration of bone tissue *in vitro*. By designing this composite network, we hope to create a composite hydrogel with enhanced adsorption, swelling, mechanical and biodegradability properties.

Nanohydroxyapatite (nHA) particles were prepared using hydrothermal method. The prepared nHA particles were characterized using FTIR, TEM, EDX and XRD. The EDX results showed that nHA was successfully prepared and its chemical structure confirmed by FTIR results. Also, the results showed that the particles obtained exhibited a hexagonal nano-rod structure with 64.8 nm length and 27.7 nm widths.

Various grades of starch grafted with acrylic acid (st-g-AA) composite hydrogels were prepared and incorporation of nHA particles with the optimal st-g-AA hydrogel was studied. The various prepared grades of st-g-AA composite hydrogels were characterized using SEM, mechanical studies, swelling ratio studies, porosity and *in vitro* biodegradation as well as *in vitro* biomineralization to confirm bioactivity of scaffold. Proposed mechanism for preparation of starch grafted with acrylic acid by microwave using bisacrylamide crosslinker was illustrated.

It was found that the swelling ratio and water uptake of the hydrogels increased with increasing the concentration of acrylic acid in St-g-AA. This may be due to increasing the number of hydrophilic groups (COOH) which are responsible for high swelling and water absorption.

It can be also seen that the porosity of the hydrogels increased with increasing the concentration of acrylic acid in St-g-AA. This may be due to an increase in hydrogel solution viscosity that prevents the escaping of bubbles from the solution. These bubbles are in turn increased the porosity due to the creation of interconnected channels. On the other hand, the higher acrylic acid concentration generally leads to very soft hydrogels with poor mechanical properties.

The results showed also a lower average degradation rate of the hydrogels in phosphate buffer solution on the 7th day with increasing the concentration of acrylic acid followed by a higher degradation rate on the 14th day.

Concerning the effect of crosslinker concentration (BAM) present in St-g-AA, it can be seen that increasing the percentage of BAM from 2 to 5% at constant acrylic acid concentration (78.4%) leads to an increase in both swelling ratio and water uptake of the hydrogels. This may be due to the increase in crosslinking density in St-g-AA hydrogels that enhances the interaction between acrylic and polymer network which is held together by covalent bonds in the produced hydrogel. Also, the porosity of the hydrogels decreased

with increasing the percentage of BAM in St-g-AA while the mechanical properties increased.

It was found also that the degradation rate of the hydrogel samples decreased with increasing the percentage of BAM on the 7th and 14th days due to increase the holding of polymer chains together by covalent bonds.

Additionally, the results showed the effect of incorporation of nHA into St-g-AA (1:2.5) hydrogel at constant percentage of crosslinker (3%) on the swelling ratio, water uptake, and porosity of hydrogel samples. It can be seen that nHA particles reduced the swelling ratio, water uptake, and porosity of St-g-AA hydrogel. This reduction may be due to the presence of nHA particles that occupy the empty voids within the polymeric scaffold and interact with the hydrogel. This effect also enhanced Young's modulus and ultimate strength of the hydrogel. On the other hand, slower degradation rate of St-g-AA/nHA was observed in phosphate buffer solution due to the crystalline structure of nHA particles.

nHA particles in St-g-AA hydrogel enhanced the number of nucleation sites on exposure to body fluids, resulting in increasing the surface area and providing more nucleation sites for apatite formation.