

UNIVERSITI TEKNOLOGI MARA

***IN VITRO* BIOMINERALISATION
OF CALCIUM PHOSPHATE BASED
SYNTHETIC BONE GRAFTING
MATERIAL USING
POLY(ϵ -CAPROLACTONE)
ELECTROSPUN FIBRE MEMBRANE**

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MSc

September 2021

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.


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Thesis Title : In Vitro Biomineralisation of Calcium Phosphate
Based Synthetic Bone Grafting Material Using
Poly(ϵ -caprolactone) Electrospun Fibre Membrane

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ABSTRACT

Bone autografts and allografts are the most frequently employed methods for bone replacement in orthopaedic surgeries. However, the effectiveness of autografts is severely limited, attributed to the lack of transplantable bone, complicated surgical procedure needed for the bone harvest, and donor site complications. Allografts may have introduced the additional risks of immune injection or disease transmission. Therefore, there is a critical demand for synthetic bone graft substitutes to overcome the shortcomings of bone grafting. A versatile, synthetic matrix material for bone regeneration is engineered using electrospun fibres. In this work, the electrospinning parameters were controlled to produce favourable porous fibre that can aid in forming calcium phosphate during an *in vitro* biomineralisation process. The parameters varied in flow rates (0.05, 0.10, 0.15, and 0.20 ml/min) and distances of the needle tip-to-the collector (10 and 15 cm), for each flow rate. The characterisation of the PCL electrospun fibre membrane was observed using FESEM, ImageJ, mercury porosimeter, and digimatic micrometre gauge, to investigate the fibre's morphological structure, fibre diameter, fibre pore diameter distribution, and membrane thickness. The effects of the electrospinning parameters were discussed and the PCL electrospun fibre membrane was incubated for *in vitro* biomineralisation process. The study found PCL electrospun fibre membrane at a flow rate of 0.10 ml/min and 15 cm distance of the needle tip-to-the collector are the optimum conditions to produce the membrane. The resultant membrane exhibited a large pore diameter (10 μm) uniform thick layer (0.363 ± 0.039 mm) that can be easily peeled off from the aluminium foil on the collector. Then, the membrane underwent the bioactivity process using different concentrations of simulated body fluid (SBF) (1.0, 1.5, and 3.0 times) at different temperatures (37 and 25 $^{\circ}\text{C}$) for 7, 14, and 21 incubation days to promote the precipitation of the calcium phosphate. The results showed that the formation of calcium phosphate within the fibre membrane at body temperature and room temperature. From SEM analysis, a ball-shaped apatite structure was formed with an apatite diameter of 1.8 μm . The calcium phosphate was produced at the surface and in the pores of the PCL electrospun fibre membrane. The EDX analysis shows that by increasing the incubation days to 21, the resultant crystal apatite formed decreased at body temperature (37 $^{\circ}\text{C}$). The estimated amount of calcium (Ca) decreased from 2.11 to 0.30 wt%, whereas the amount of phosphorus (P) reduced from 1.94 to 1.60 wt% at SBF 3.0 x. However, at room temperature (25 $^{\circ}\text{C}$), more crystal apatite was formed as the incubation days increased. The estimated amount of Ca increased from 1.31 to 5.58 wt%, whereas the amount of P increased from 1.77 to 3.58 wt% at SBF 3.0 x. The EDX results proved there were elements of calcium (Ca) and phosphorous (P). The XRD analysis shows the presence of bone apatite on the PCL electrospun fibre membrane. The current study suggested that the bioactivity of PCL electrospun fibre membrane at body temperature could be enhanced by modifying the morphological structures of PCL in order to improve the adherence of apatite on PCL. From the experimental results, the current study has contributed to a new piece of knowledge on the importance of fibre membrane to be employed in cell proliferation, migration, and differentiation for biomineral application, especially in bone regeneration.

ACKNOWLEDGEMENT

Firstly, I wish to thank God for giving me the opportunity to embark on my MSc and for completing this long and challenging journey successfully. My gratitude and thanks go to my supervisor Ts. Dr. Nor Dalila binti Nor Affandi and Dr. Nurdiana binti Samsulrizal.

My appreciation goes to all the laboratory staff and technicians who provided the facilities and assistance during sampling. Special thanks to my friends for supporting me with this project.

Finally, this thesis is dedicated to the unlimited support of my very dear father and mother for the vision and determination to educate me. This piece of victory is dedicated to both of you. Alhamdulillah.

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