Development of a Soft Sensor to Predict the Molecular Weight of Polylactide (PLA) in a Melt Extrusion Process

DEVELOPMENT OF A SOFT SENSOR TO PREDICT THE MOLECULAR WEIGHT OF POLYLACTIDE (PLA) IN A MELT EXTRUSION PROCESS

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INTRODUCTION

Polylactide (PLA) offers advantages over traditional materials for implantable medical devices such as sutures and fixation screws. PLA is bioresorbable which eliminates the need for a second operation to remove any device[1]. Ideally a PLA medical implant will provide mechanical support for a desired time frame while transferring the load gradually at a controlled rate to the body's recovering bone and soft tissue during the regeneration[2]. Medical grade PLA is a high cost commodity costing up to €5000/kg. Finding optimised melt processing conditions is generally based on trial and error and is very sensitive to batch to batch variations. Product characterisation is lab based and offline meaning full production runs can be completed while the manufacturer has no knowledge on whether the product is in or out of spec. As a result, typical scrap rates of a PLA medical grade product can be up to 25-30%. Molecular weight (Mw) is viewed as a key indicator of the end product quality and is typically characterised offline by Gel Permeation Chromatography (GPC). This work aims to develop a soft sensor model which can provide real time feedback on the changes to Mw during melt processing. This will reduce manufacturing costs and minimise the number of rejected samples.

MATERIALS AND METHODS

A medical grade PURASORB Polylactide copolymer having L/D ratio of 96/4 (PLDLA 96/4) was obtained from Purac Biochem bv. A slit die was designed for addition to a twin screw extruder which forms part of a fibre melt spinning process. The pressure drop measurement along the slit die channel together with knowledge of the throughput and the slit channel geometric constants can be used to estimate the shear viscosity. A number of different melt processing conditions were investigated using varying temperature profiles and varying feed rates. The shear viscosity estimates required compensation for the effect of different melt temperatures and shear rates at the different process conditions. An Arrhenius model was investigated for temperature compensation and a power law model used for shear rate compensation. Offline rheological testing was carried out to determine the modelling constants. The correlation between Mw characterised by GPC and the estimated shear viscosity from in-process measurements was investigated.

RESULTS & DISCUSSION

Correlation was found between the shear viscosity estimations and the characterised Mw of the extruded fibre (*Figure 1*). There are some outliers which are considered to be due to the modelling constants used to compensate the shear viscosity estimates. Further offline rheology will need to be completed to investigate additional shear rates and temperatures to improve the existing model. This in itself is inherently difficult as the material also tends to degrade during this offline testing. Future work will look at improving the rheology test method to try to minimise any degradation prior to the onset of collecting test data. Further, Mw was assessed on a small sample batch produced during each production run. This is not necessarily representative of the Mw of the product over the entire production run. This may have influenced the Mw predictions.

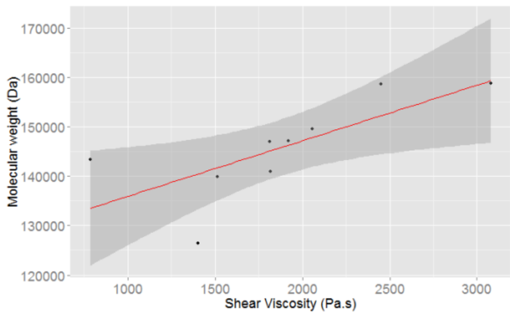

Figure 1 Shear viscosity is plotted against Mw for all processing conditions. A line of best fit is plotted and a confidence interval of 0.95 is highlighted by the greyed region.

However, this initial work shows potential for a low cost 'soft sensor' to be developed which can be retrofitted to conventional extrusion machinery to optimise melt processing of a medical grade PLA.

REFERENCES

Cutright *et al.*, Oral Surgery, Oral Medicine, Oral Pathology 31:134-139, 1971
Brown and Farrar, Plastics, Rubber and Composites 37:46-49, 2008