Master's Thesis Engineering Technology

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Optimisation and characterisation of nano-hydroxyapatite/polylactide composites using Fused Deposition Modelling technology

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Introduction

In Fused Deposition Modelling (FDM) processes, neither the materials nor the process have been studied in a systematic manner towards functional components production. The aim of this study was the systematic characterisation of the influence of the printing speed and the printing temperature on the mechanical properties of a new nano-hydroxyapatite polylactide composite.

Materials & Processing

Figure 1 PLE 005 granules (bottom) and aqueous hydroxyapatite paste (top).

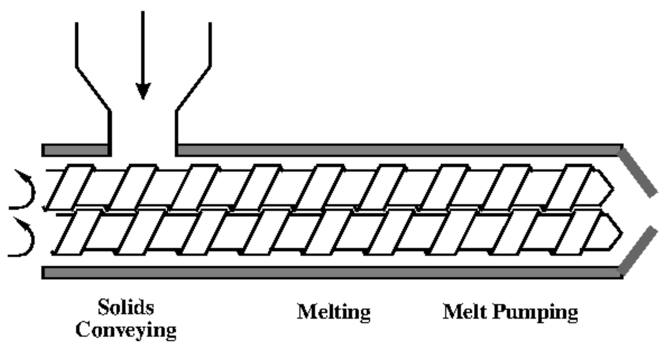

Figure 2 Twin-screw extruder [1].

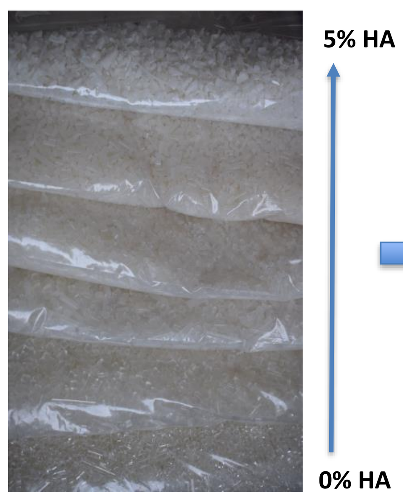

Figure 3 Pellets obtained after twin-screw extrusion.

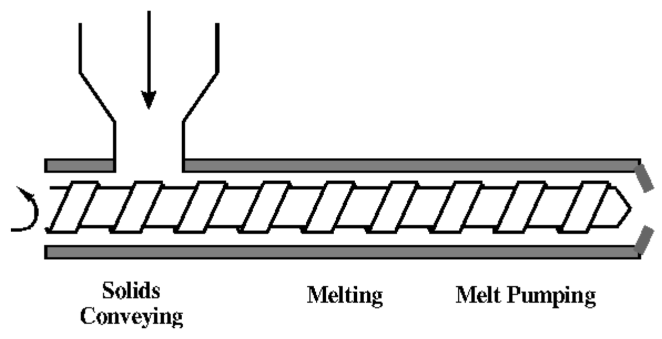

Figure 4 Single-screw extruder [2].

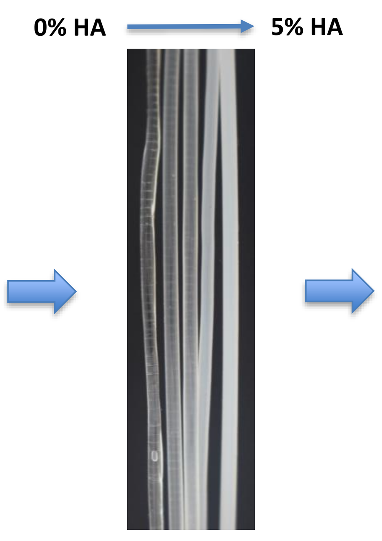

Figure 5 Filaments obtained after single-screw extrusion.

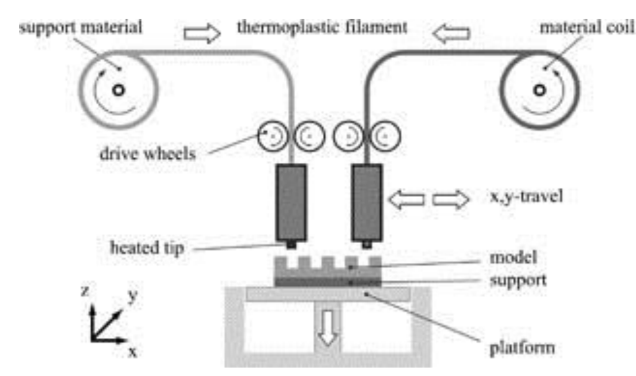

Figure 6 Fused deposition modelling printer [3].

Hydroxyapatite (HA) nanoparticles were added to polylactide (PLA) during a twin-screw extrusion (Fig. 1 & 2) in 0; 0,5%; 1%; 3% and 5 wt.%. After extrusion, the obtained PLA/nHA granules were extruded with a single-screw extruder (Fig. 3 & 4) to obtain filaments for the FDM printing process (Fig. 5 & 6). The filaments were used to create dynamic mechanical analysis (DMA) and tensile test specimens.

Results

TGA, DSC, Oscillatory rheology, DMA, Tensile tests, permeability tests, TEM and XRD analysis were used for the characterisation of the PLA/nHA composites.

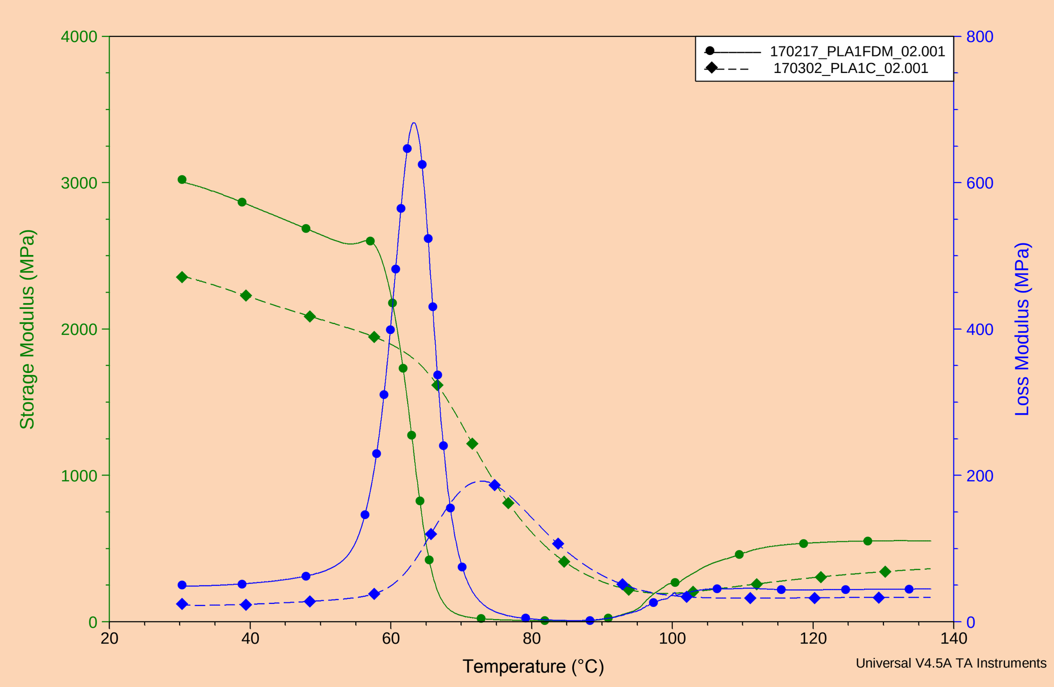

Figure 7 Storage modulus and loss modulus of compression moulded and FDM printed PLA/nHA 1% samples.

DMA showed that the storage modulus of FDM printed samples was higher than that of annealed compression moulded granules, which was surprising (Figure 7). It was suggested that this was due to an alignment of polymer chains caused by the FDM process, but additional testing is required.

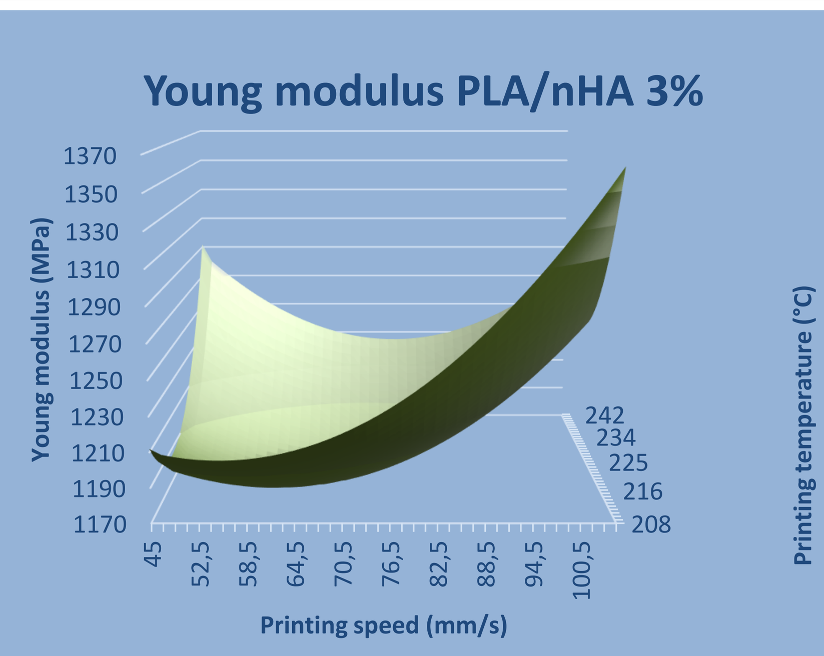

Figure 8 Doehlert response surface of the Young modulus of PLA/nHA 3%.

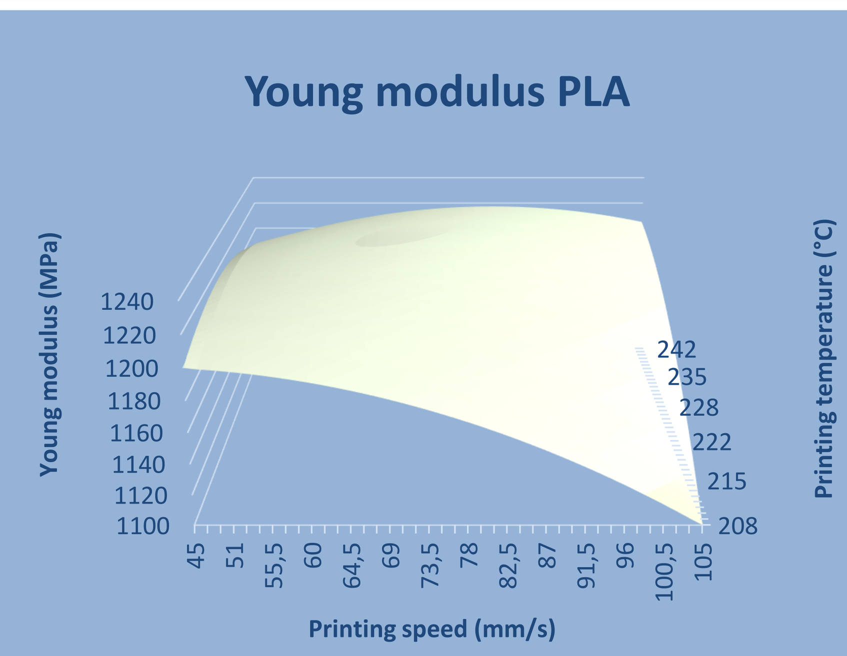

Figure 9 Doehlert response surface of the Young modulus of PLA.

The results of the tensile tests of FDM printed samples were used to create response surfaces in function of the printing parameters, by applying Doehlert Response Surface Methodology (Fig. 8 and 9) [4]. For PLA/nHA 3% a minimum was obtained within the domain, while for pure PLA a maximum was obtained in the investigated domain. For the other formulations, the Doehlert model did not correspond to actual tensile responses, due to a more complex behaviour.

$$y_{th} = a_0 + a_1.x_1 + a_2.x_2 + a_{12}.x_1.x_2 + a_{11}.x_1^2 + a_{22}.x_2^2 + \varepsilon$$

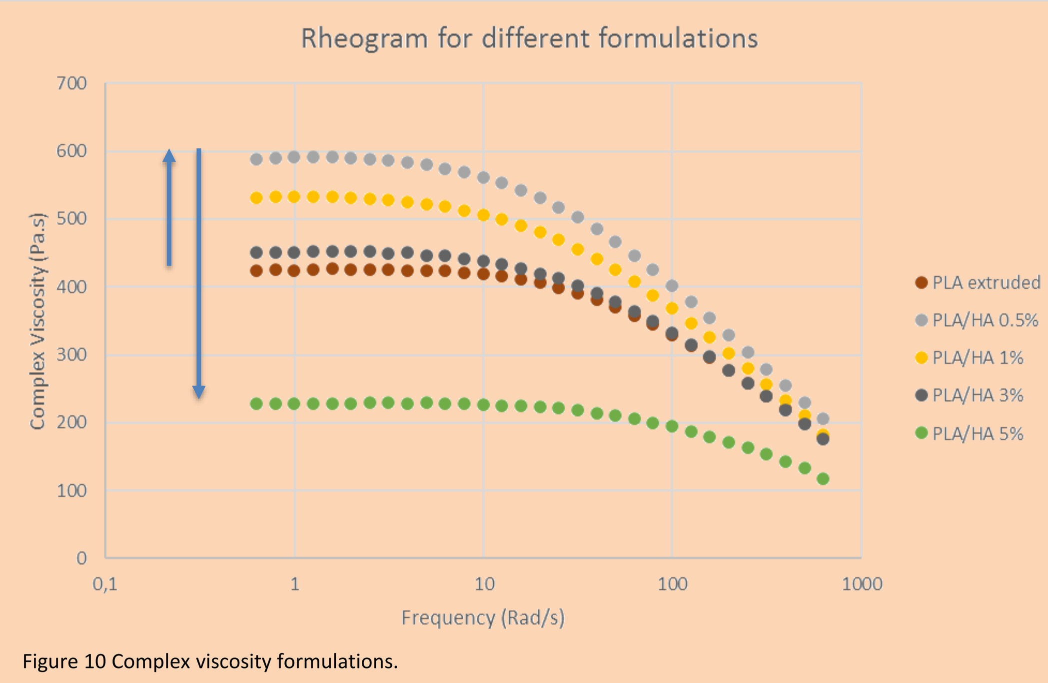

Figure 10 Complex viscosity formulations.

The complex viscosity of the composites was modelled using the Carreau-Yasuda model (Fig. 10) [5]. The first addition of HA resulted in a thickening effect that decreased when more HA was added; and for 5% HA a thinning effect appeared

$$\eta(\dot{\gamma}) = \eta_0[1 + (\lambda\dot{\gamma})^a]^{\frac{n-1}{a}}$$

R^2> 0,999

Conclusion

In this study PLA/HA nanocomposites were successfully printed using FDM. Doehlert response surface methodology showed that there was a strong influence of the printing parameters on the tensile properties and a strong interaction between the printing speed and printing temperature. The dynamic mechanical analysis showed a higher storage modulus for the FDM printed samples. Rheological analysis showed that higher HA concentrations did not result in a higher viscosity, which could have prevented printing.

References

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