

Master of Science HES-SO in Life Sciences

Development and application for reinforced plastic-microparticle composite material for polymer injection

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CHEMICAL DEVELOPMENT & PRODUCTION

HEIA-FR

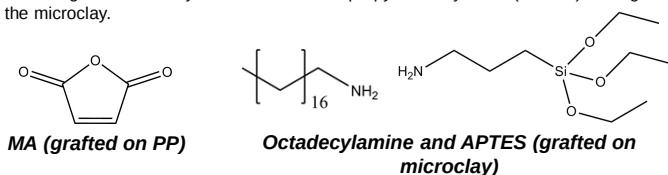
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DESCRIPTION

The synthesis of reinforced polymers with particles, also known as composites, expands the scope of plastics use. Composites have the properties of being stiff due to the microparticles and at the same time ductile due to the polymer matrix. These two properties allow for molding plastic pieces that are resistant to impacts but at the same time absorb shocks through elastic deformations.

Microcomposites also show interesting advantages over other working materials like glass or metal. They have a low price and easy-to-shape properties. Furthermore, many different polymer grades with different features are commercialized and the small size and round shape of the microparticles permit to mold thin and small plastic pieces.

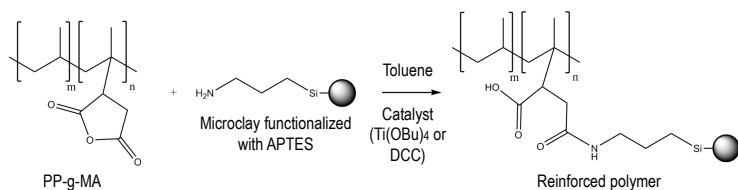
In this project, composites of polypropylene (PP) reinforced with microclay particles have been fabricated. However, these two products have opposite chemical properties and they tend to form separate phases. To overcome this issue, functionalizations under the form of organic molecules are grafted on both products. Maleic anhydride (MA) was grafted on PP to form PP-g-MA. Octadecylamine and 3-aminopropyltriethoxysilane (APTES) were grafted on the microclay.



OBJECTIVES

The main objective of this Master Thesis is to synthesize a reinforced polymer made of PP and microclay which is mechanically stiff and elastic, homogenous and free of delamination.

An amidation reaction is performed between MA and APTES to covalently bond the microclay to PP. The amino group on APTES reacts with the carbonyl groups on MA.



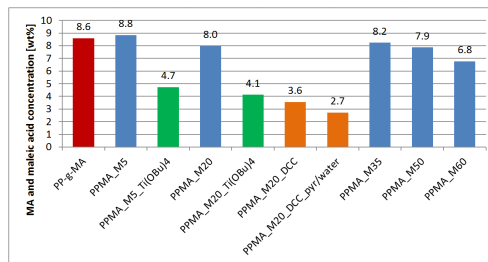
Amidation reaction to synthesize a reinforced polymer

The amidation reaction is optimized by using a catalyst to avoid the incorporation of an excess of microclay in the microcomposite since the clay is more expensive than PP-g-MA. Also the agglomeration of the microclay can lead to mechanical defects in the final product. The progress of the reaction is analysed through the evolution of MA (and maleic acid formed by the hydrolysis of MA) quantity which decreases when it reacts with the amino groups on the microclay.

RESULTS

FTIR was identified as an appropriate method to follow the evolution of the MA and maleic acid concentration. Both products show different peaks in the FTIR spectrum which are used for their quantification based on a calibration curve. PP-g-MA used in this project contains 8.6 wt% of MA. The decrease of this quantity indicates that the amidation reaction successfully took place.

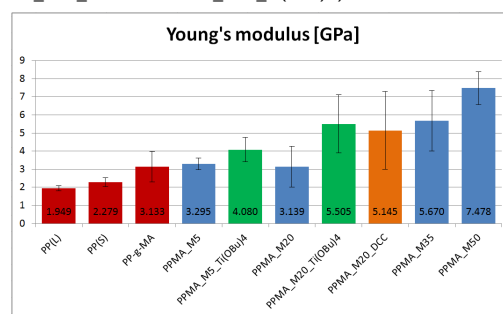
20 wt% of microclay incorporated in PP-g-MA and the use of N,N'-Dicyclohexylcarbodiimide (DCC) as catalyst have shown the best result. PP-g-MA was treated first with pyridine in water to hydrolyze the MA molecules allowing the formed maleic acids to react more effectively with the microclay (PPMA_M20_DCC_pyr/water). Only 2.7 wt% of MA and maleic acid remain in the final product.



Evolution of MA and maleic acid concentrations in the composites (contain various wt% of microclay indicated as M)

The reinforcement of the composite is measured by nanoindentation. The Young's modulus value representing the stiffness of the material shows an increase with the quantity of stiff microclay incorporated. An increase is also observed when a catalyst is used to perform the amidation reaction for the same quantity of microclay.

The highest reinforcement for a low microclay content is achieved with 20 wt% of functionalized microclay in PP-g-MA using DCC or Ti(OBu)₄ as catalysts for the amidation reaction (PPMA_M20_DCC and PPMA_M20_Ti(OBu)₄).



Nanoindentation measurement of the composites (PP(L) has long carbon chains and PP(S) has small chains)

CONCLUSION

PP-g-MA was successfully reinforced with microclay by performing an amidation reaction. The reaction was optimized by using pyridine and water to transform MA in maleic acid. Then DCC was used as catalyst to do the amidation reaction resulting in the formation of a covalent bond between PP-g-MA and the microclay. This link between the two products leads to an increase in the Young's modulus from 1.95 GPa (pure PP) to 5.14 GPa as observed for an incorporation of 20 wt% of microclay. This corresponds to an increase of 160% for the Young's modulus.

However, this reinforced composite is brittle due to PP-g-MA short carbon chains. Therefore, this material is currently not adapted for an extrusion and/or injection process to form plastic pieces. To overcome this issue, it could be mixed with a PP having longer carbon chains.