

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

Virginie Kottelat, Thierry Chappuis, ChemTech  
Stefan Hengsberger, iRAP

## 1. Introduction

Composites made of polypropylene (PP) in which micrometer size clay particles are incorporated are synthesized via solution blending. The microparticles stiffness and the ductile polymer matrix permit to mold small plastic pieces that are resistant to impacts but also absorb shocks through elastic deformations.

However, PP and microclay particles have opposite chemical properties and they tend to form separate phases. To allow their mixing, functionalizations under the form of organic molecules are grafted on both products. Maleic anhydride (MA) was grafted on PP with short carbon chains to form PP-g-MA. Octadecylamine and 3-aminopropyl-triethoxysilane (APTES) were grafted on the microclay. The MA groups on PP react via an amidation reaction with the amino groups on the microclay to form chemical bonds that link strongly the polymer to the microparticles.

The high price of microclay and their tendency to agglomerate (which leads to mechanical defects) motivate to incorporate only small amounts in the composite. A way to achieve a stiff composite with lower amounts of microclay consists in forming more chemical bonds between PP-g-MA and the microclay. For this study, 8.6 wt% of MA is grafted on PP so theoretically, only 20 wt% of the functionalized microclay is sufficient to react with all MA.

## 2. Results

### 2.1. Optimization of the chemical linkage

The amidation reaction between PP-g-MA and the microclay is optimized by using a catalyst like N,N'-dicyclohexylcarbodiimide (DCC) or titanium butoxide ( $Ti(OBu)_4$ ). 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 reacting with the functionalizations on the microclay.

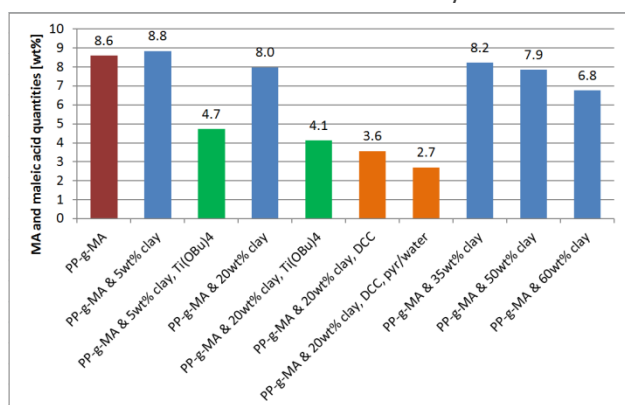


Figure 1 : MA and maleic acids evolution depending on microclay content and the use of a catalyst

The incorporation of 20 wt% of microclay in PP-g-MA and the use of DCC as catalyst have shown the greatest decrease of MA and maleic acid quantities; only 2.7 wt% of the functional molecules have not reacted (see Figure 1). PP-g-MA was treated first with pyridine in water to hydrolyze the MA molecules into maleic acids, which react more efficiently with the microclay functionalizations.

### 2.2. Mechanical reinforcement

The Young's modulus and hardness of the composite is determined by nanoindentation. The stiffness of the composite shows a correlation with the quantity of incorporated microclay but also with the catalyst used to improve the amidation reaction.

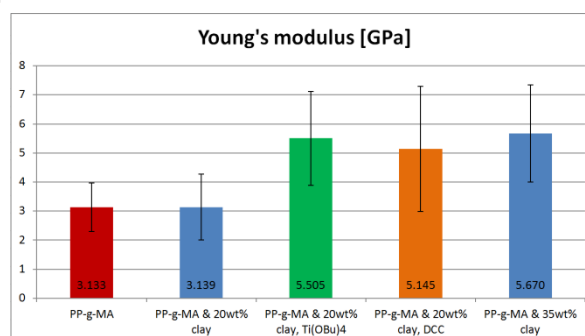


Figure 2 : Stiffness of the composite improved with the optimisation of the amidation reaction

Stiffness increases of 76% and 64% (compared to PP-g-MA) are observed when  $Ti(OBu)_4$  and DCC are respectively used as catalysts for 20 wt% of microclay incorporated in PP-g-MA (see Figure 2). These results are closed to the 81% obtained for 35 wt% of microclay when no catalyst is used.

## 3. Conclusion

PP-g-MA is successfully reinforced with microclay by performing an amidation reaction between their respective functional molecules. The reaction is optimized by using pyridine in water to transform MA in maleic acid. Then DCC is used as catalyst for the amidation reaction resulting in the formation of strong chemical bonds between PP-g-MA and the microclay. This optimization is equivalent in stiffness to introducing more clay in the composite but it is cheaper and avoids product defects due to microclay agglomeration

However, this stiff composite is brittle due to the low intercalation of the PP-g-MA short carbon chains. This issue can be overcome by adding PP with longer carbon chains that acts as a matrix around the small carbon chains to avoid their delamination.