



DETERMINATION OF GRAFTING DEGREE OF MALEIC ANHYDRIDE ON PP

The aim of this report is, to show that our Methods of Grafting Degree Determination are versatile and accurate.

MATERIALS USED

For this report, we used scrap PP that was grafted with MAH at Graft Polymer. Matrix material was provided by Customer.



TESTS PERFORMED

For the determination of grafting degree, we used:

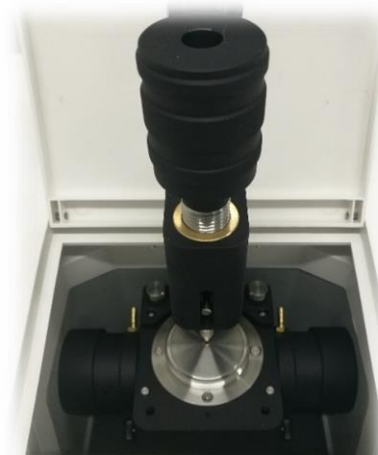
- Fourier Transformation Infrared Spectrometer (FTIR, picture on right)
- Back Titration with KOH and HCl

We tested two samples:

- Stored in LDPE bags, as prepared – FTIR Only
- Chemically purified samples -

Purification was performed in two steps:

- Dissolving grafted polymer
- Elimination of unreacted MAH



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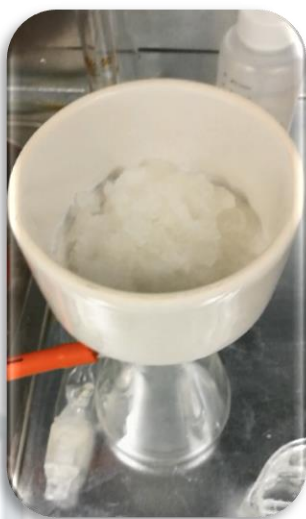
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PROCESS



We dissolve 5 gr of PP-g-MAH in boiling xylene (Isomeric mixture) in a reflux (left)
After that we let it cool (right)



The solution is precipitated using acetone – acetone also dissolves unreacted MAH away (left)

The sample is dried in a fume hood for 3 hours and in an oven at 60°C for 2 hours (right)

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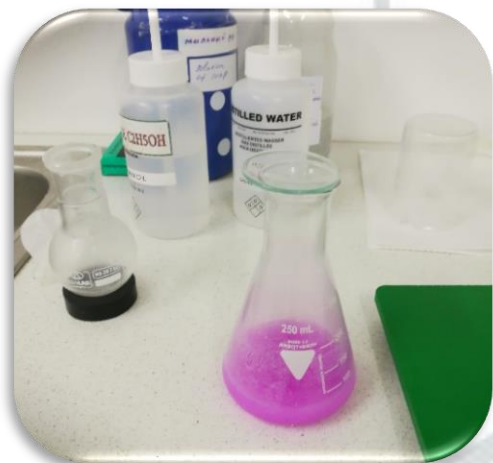
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After the sample is dried, we dissolve 1 g again in boiling xylene and add 5 mL of the base KOH (0,1 M)

Reaction with anhydride and KOH occurs, using up all anhydride groups

We added KOH in excess, in order to titrate it with HCl



We add 2 drops of the indicator phenolphthalein to the solution – it is pink because the solution is basic

We titrate the leftover KOH with HCl (0,05 M)

Titration is finished when the solution is colorless

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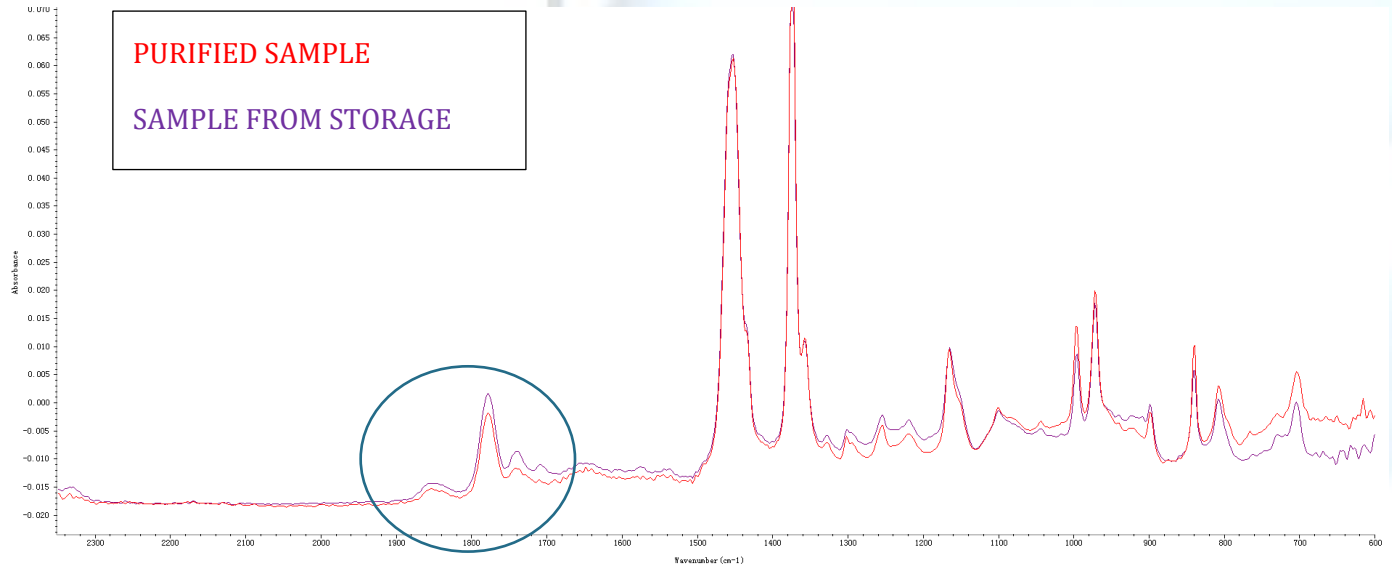
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RESULTS

FTIR RESULTS



On the above picture, we can see the comparison between “raw” (Purple spectra) and purified (Red spectra) samples. Maleic anhydride is seen in the area of 1850 cm^{-1} and 1780 cm^{-1} , according to the x axis. The relevant area is circled.

- Calculated MAH on the sample from storage is: 1,97% MAH (Including unreacted MAH)
- Calculated MAH on the purified sample is: 1,46% MAH (No unreacted MAH)

TITRATION RESULTS

For the determination of grafting degree, we used the following formula:

$$GD (MAH) = \frac{c(KOH) \cdot V(KOH \text{ leftover}) \cdot M(MAH)}{m(\text{sample})} = 1,598\%$$

$c(KOH)$ – KOH Concentration [mol/L]

$V(KOH \text{ leftover})$ - KOH, that wasn't used for neutralizing HCl [L]

$M(MAH)$ – Molar mass of grafted MAH [=99,07 g/mol]

$m(\text{sample})$ – mass of the purified polymer sample [g]

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CONCLUSIONS

- Grafting degree, measured with FTIR before purification is higher than that of purified sample, which means that 0,51% of MAH was unreacted
- By purifying the sample, we determined the grafting degree of the sample to be around 1,5%
- The difference between the methods is only around 0,138%
- When increasing the number of samples, the deviation grows smaller
- FTIR and titration are COMPARABLE METHODS



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