
PROJECT 7:

THERMO-REVERSIBLE CROSS-LINKED
NANOCOMPOSITE MATERIALS BASED ON EPM
RUBBER AND 3-(1-TRI-ETH-OXY-SILYL-
PROPYL) MALEIMIDE

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ABSTRACT

Thermo-reversible polymers are becoming increasingly important, due to their potential recyclability. Nano-composite materials are, also, increasingly being employed as the: mechanical, electro-physical, optical and, even, magnetic properties can be modified through the adjustment of the nanoparticle filler size. These materials tend not be recyclable, however if the nanoparticle filler were cross-linked, using a Diels-Alder reaction, the resulting polymer should be thermo-reversible and, potentially, recyclable. To crosslink a modified EPM rubber (Polymer A) two reactions were performed: a Diels-Alder reaction and a hydrolysis/condensation of the hydrophilic ends of 3-(1-triethoxysilylpropyl)maleimide. Two polymers were synthesised: one where the Diels-Alder reaction was performed last (Polymer B) and one where the Diels-Alder reaction was performed first (Polymer C). IR spectroscopy and EDS of polymers B and C confirmed that crosslinking was successful in both polymers. The tensile tests of Polymer C consistently displayed a lower Young's modulus and higher ultimate stress, relative to Polymer B, with both demonstrating higher Young's moduli and ultimate stresses, relative to Polymer A. SEM images suggested that the size and distribution of the silica nanoparticles in Polymer C were smaller and more uniform than those present in Polymer B. Performing the Diels-Alder reaction first appeared to yield a polymer with superior tensile properties, relative to a polymer where the Diels-Alder reaction was performed last.

INTRODUCTION

A thermo-reversible polymer is a polymer, which, under a certain temperature, breaks down to its composite compounds. This makes it possible to recycle. This thermo-reversible property is, however, uncommon in reinforced composite materials, as the creation of such a material involves a filler surface being covalently bonded to a polymer matrix, for the purpose of strengthening the original polymer; typically this bonding type is not thermo-reversible. One possible method to attain this thermo-reversible property, for a reinforced composite material, is to bond a filler surface to a polymer matrix, by a thermo-reversible reaction, in this case, a Diels-Alder reaction.^[1] Furthermore, through the utilisation of silica nanoparticles a nano-composite polymer can be synthesized. These materials are becoming increasingly important as the: mechanical, electro-physical, optical and, even, magnetic properties, of the resulting polymer can be tailored to its function, by controlling the size of the nanoparticle filler.^[6] By considering this property of nano filler molecules alongside the thermo-reversible nature of the Diels-Alder bond it is obvious that research into the combination of these two facets of polymer chemistry will become more important, in the future, as alternative, recyclable materials are sought after to reduce pollution.^[3]

THEORY

THE POLYMER

Ethylene propylene rubber (EPM rubber) is polymer that is widely used: to produce seals and as an insulator, for high voltage cables, due to its chemical and physical properties. e.g. resistance to: heat, oxidation, ozone, the weather, and colour loss, while, also, being an electrical insulator.^[4]

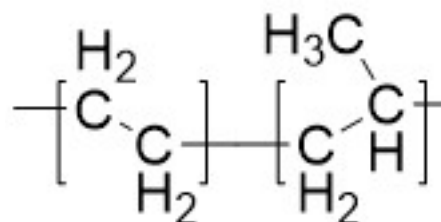


Figure 1: The Structure of EPM Rubber [4]

It is, however, obvious from this structure that modification is near impossible, as there are no active sites, which in turn gift the molecule its multitudinous resistances. For this reason an, already modified polymer was further modified contain a functional group, where crosslinking could be performed.

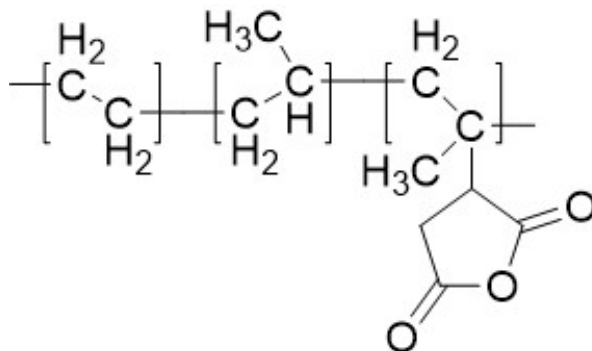


Figure 2: The modified EPM rubber

This modified EPM rubber was further modified to contain a site, where a Diels Alder reaction could be possible; a conjugated diene was formed. This is done through a nucleophilic ring opening of the cyclic anhydride functional group of the modified EPM polymer, with 2-Aminomethylfuran.

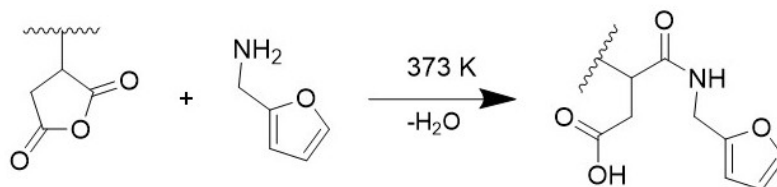


Figure 3: Nucleophilic ring opening of the cyclic anhydride functional group of the EPM polymer with 2-Aminomethylfuran

The resultant 4-((2-Furylmethyl)amino)-4-oxobutanoic acid functional group must then undergo a cyclic amination to form a 1-(2-Furylmethyl)-2,5-pyrrolidinedione functional group to prevent any undesired reactions the carboxyl group could initiate, specifically with the hydroxyl groups of 3-(1-trihydroxysilylpropyl)maleimide, throughout the crosslinking process.

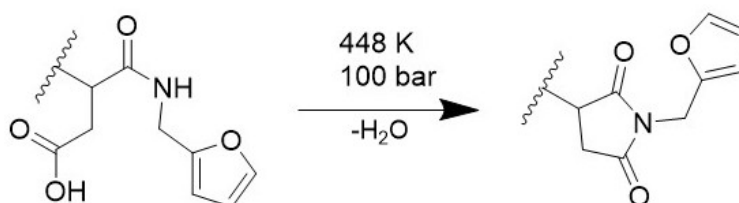


Figure 4: Cyclic Amination of the 4-((2-Furylmethyl)amino)-4-oxobutanoic acid functional group to 1-(2-Furylmethyl)-2,5-pyrrolidinedione

(This polymer will be referred to as "Polymer A" throughout the rest of this report for convince.)

THE FILLER MOLECULE

3-(1-triethoxysilylpropyl)maleimide is an inorganic silica molecule, which will be hydrolysed to 3-(1-trihydroxysilylpropyl)maleimide for the reaction to function.

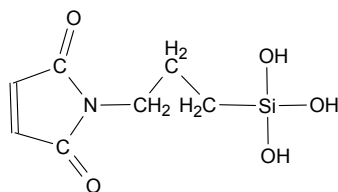


Figure 5: Chemical Structure of 3-(1-trihydroxysilylpropyl)maleimide

This hydrolysis results in the formation of a hydrophilic “head” of hydroxyl groups around the silica atom. These alcohol groups can then condense to form Si-O-Si bonds, with adjacent maleimide molecules, forming a “mono-disperse spherical silica material”, or nanoparticle filler.^[5]

HYPOTHESIS

The method whereby a silica particle is first synthesised then cross-linked into the EPM rubber should lead to a polymer with differing properties, relative to its counterpart which was formed inversely. This is because when the 3-(1-triethoxysilylpropyl)maleimide is bonded to the modified EPM rubber the subsequent hydrolysis and condensation, crosslinking the polymer, is expected to hinder the hydrophilic hydroxysilyl ends from forming a mono-disperse spherical silica material and should instead simply crosslink one end of a polymer molecule to another 3-(1-trihydroxysilylpropyl)maleimide end of another polymer chain. Performing the hydrolysis/condensation before performing the Diels Alder reaction results in the formation of silica nanoparticles. These larger spherical molecules should crosslink the polymer less effectively, due to their size, leading to a weaker material.

CROSSLINKING 3-(1-TRIETHOXSILYLPROPYL)MALEIMIDE TO THE EPM RUBBER

Two general pathways are, therefore, available to crosslink EPM rubber to 3-(1-trihydroxysilylpropyl)maleimide:

One where 3-(1-triethoxysilylpropyl)maleimide is first hydrolysed, then condensed to form silica nanoparticle filler molecules and finally undergoes a Diels Alder reaction, with the EPM rubber, thereby bonding the nanoparticle filler to the polymer; one of the other double bonds of this nanoparticle will then, also, bond to another EPM rubber chain, effectively crosslinking the polymer.

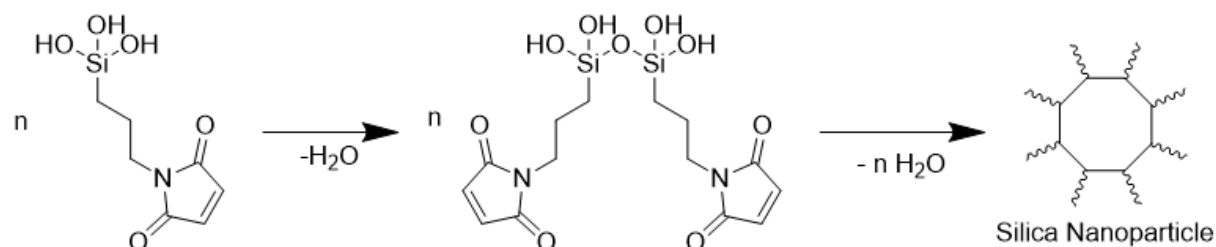


Figure 6: Silica Nanoparticle formation through condensation of oxysilyl ends

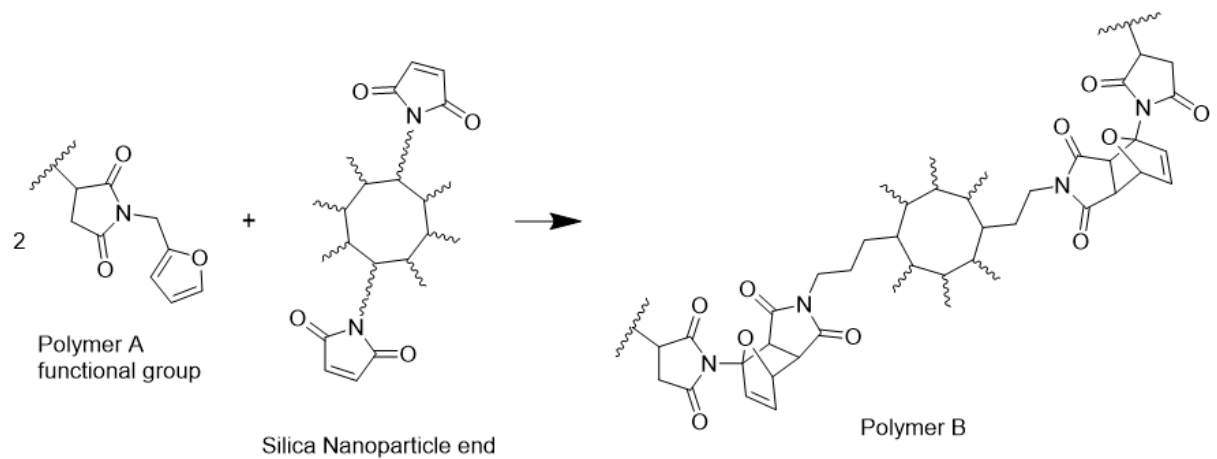


Figure 7: Crosslinking of Polymer A, with Silica Nanoparticle filler, via Diels Alder reaction

This is possible because the “tail” ends of the silica nanoparticle continue to contain the maleimide structure, which contains a double bond. These double bonds are used in a Diels Alders reaction, with the conjugated diene from the EPM rubber, thereby crosslinking the EPM rubber, with the silica nanoparticle

(For convenience, the polymer formed in this manner will be referred to as “Polymer B” for the rest of the report.)

The second path way is the reverse of this; the Diels Alder reaction bonds the EPM rubber to 3-(1-triethoxysilylpropyl)maleimide, followed by the hydrolysis/condensation of the ethoxysilyl ends of the 3-(1-triethoxysilylpropyl)maleimide functional group crosslinking the polymer.

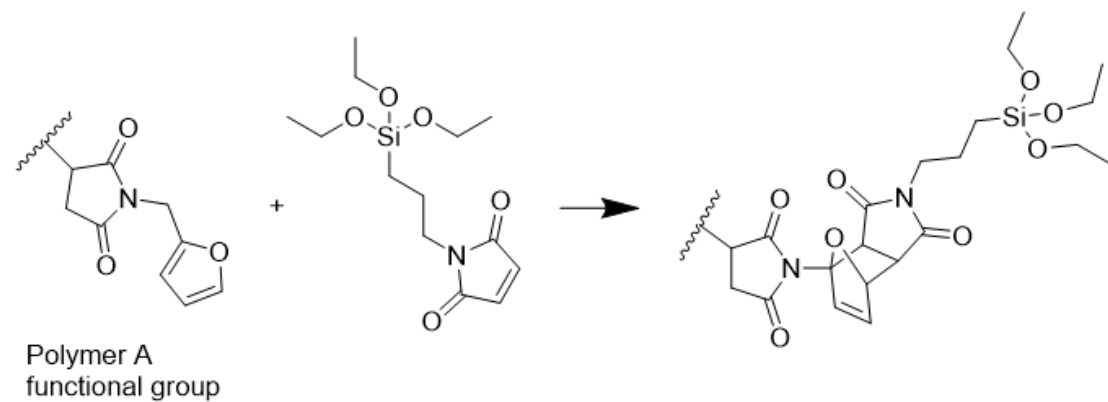


Figure 8: Bonding of 3-(1-triethoxysilylpropyl)maleimide to the Polymer A functional group, via Diels Alder reaction

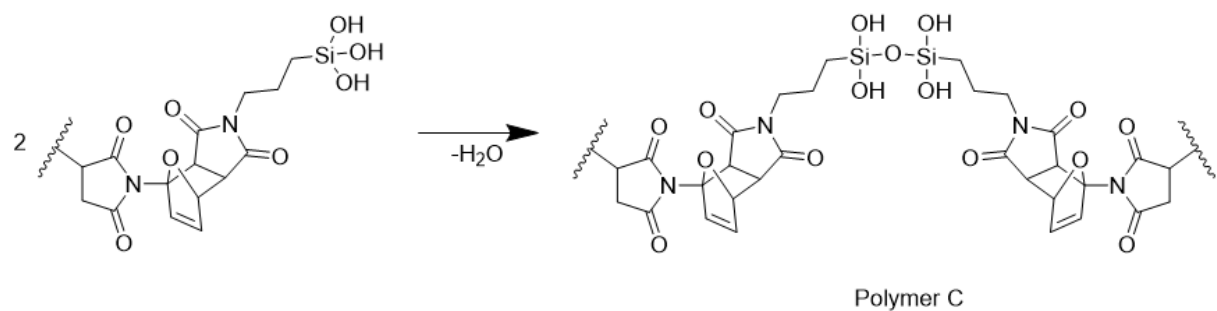


Figure 9: Crosslinking reaction to form Polymer C, via hydrolysis of oxyisilyl ends

(For convenience, the polymer formed in this manner will be referred to as “Polymer C” for the rest of the report.)

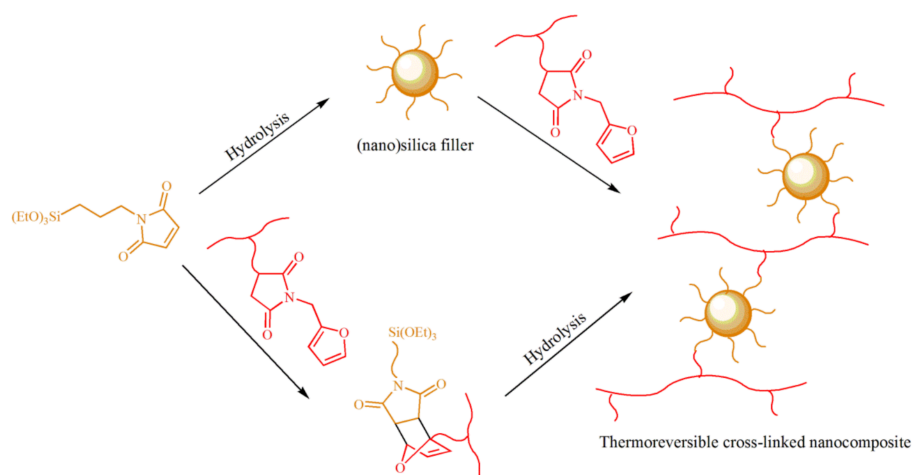


Figure 10: The two synthesis routes for crosslinking the modified EPM rubber with the silica nanoparticle

METHODOLOGY

POLYMER A: CREATION OF THE 1-(2-FURYLMETHYL)-2,5-PYRROLIDINEDIONE FUNCTIONAL GROUP

(18g) modified EPM rubber was dried for 24 hours at 175°C then placed in a brabender with 2.6g 2-Aminomethylfuran, under inert conditions. The resulting polymer was collected and exposed to a filter press set to 175°C and 100 bar for 30 minutes.

POLYMER B: THE FORMATION OF THE SILICA NANOPARTICLES THEN THE DIELS ALDER CROSSLINKING REACTION

(0.5g) 3-(1-triethoxysilylpropyl)maleimide was added to 20mL propanol, 2 drops trifluoromethylsulfonic acid and 20mL demi-water, followed by refluxing at 100°C, for 48 hours. Using a rotary evaporation the: demi-water, acid and propanol were removed, leaving the silica nanoparticles in the flask. 200mL THF with 5g Polymer A dissolved into it was then added to the flask which was refluxed at 60°C, for 24 hours. The resulting polymer was then shaped into “dogbones” for tensile testing using at press at 130°C for 15 minutes, followed by annealing at 50°C for 72 hours. An IR spectroscopy, and SEM were performed, along with 5 tensile tests.

POLYMER C: THE DIELS ALDER REACTION THEN THE HYDROLYSIS/CONDENSATION OF THE HYDROXYSILYL ENDS

(5g) Polymer A was dissolved in de-hydrolysed THF, along with (0.23g) 3-(1-triethoxysilylpropyl)maleimide and refluxed at 60°C, for 24 hours, under an argon atmosphere. A solution of: 20mL propanol, 2 drops trifluoromethylsulfonate acid and 20mL demiwater, was added to the resulting polymer solution, followed by refluxing at 100°C, for 24 hours. The resulting polymer was then shaped into “dogbones” for tensile testing using at press at 130°C for 15 minutes, followed by annealing at 50°C for 72 hours. An IR spectroscopy, and SEM were then performed, along with 5 tensile tests.

RESULTS

(Raman spectroscopy was attempted, however the polymers all exhibited fluorescence, making gathering results, using this technique, impossible.)

TENSILE TESTS

| Polymer Type | Test Number | Width (mm) | Thickness (mm) | Gage Length (mm) | Area (mm ²) | Ultimate Force (N) | Ultimate Stress (MPa) | Break Distance (mm) |
|--------------|-------------|------------|----------------|------------------|-------------------------|--------------------|-----------------------|---------------------|
| Polymer A | 1 | 4.00 | 2.36 | 20.00 | 9.44 | 4.60 | 0.487 | 87.10 |
| | 2 | 4.14 | 2.31 | 20.00 | 9.56 | 4.07 | 0.425 | 118.00 |
| | 3 | 4.20 | 2.24 | 20.00 | 9.41 | 4.33 | 0.461 | 133.00 |
| | 4 | 4.33 | 2.30 | 20.00 | 9.96 | 4.73 | 0.475 | 112.00 |
| | 5 | 4.24 | 2.29 | 20.00 | 9.71 | 4.48 | 0.459 | 96.80 |
| | Average | 4.18 | 2.30 | 20.00 | 9.62 | 4.44 | 0.461 | 109.38 |

Figure 11: Tensile test raw data for Polymer A

| Polymer Type | Test Number | Width (mm) | Thickness (mm) | Gage Length (mm) | Area (mm ²) | Ultimate Force (N) | Ultimate Stress (MPa) | Break Distance (mm) |
|--------------|-------------|------------|----------------|------------------|-------------------------|--------------------|-----------------------|---------------------|
| Polymer B | 1 | 4.08 | 2.24 | 20.00 | 9.14 | 6.27 | 0.686 | 13.80 |
| | 2 | 4.20 | 2.25 | 20.00 | 9.45 | 5.40 | 0.571 | 6.93 |
| | 3 | 4.32 | 2.35 | 20.00 | 10.15 | 4.30 | 0.424 | 6.16 |
| | 4 | 4.34 | 2.35 | 20.00 | 10.20 | 5.26 | 0.437 | 6.44 |
| | 5 | 4.39 | 2.45 | 20.00 | 10.76 | 5.30 | 0.493 | 7.85 |
| | Average | 4.27 | 2.33 | 20.00 | 9.94 | 5.31 | 0.522 | 8.24 |

Figure 12: Tensile test raw data for Polymer B

| Polymer Type | Test Number | Width (mm) | Thickness (mm) | Gage Length (mm) | Area (mm ²) | Ultimate Force (N) | Ultimate Stress (MPa) | Break Distance (mm) |
|--------------|-------------|------------|----------------|------------------|-------------------------|--------------------|-----------------------|---------------------|
| Polymer C | 1 | 4.53 | 2.15 | 20.00 | 9.74 | 7.20 | 0.739 | 15.90 |
| | 2 | 4.21 | 2.17 | 20.00 | 9.14 | 5.40 | 0.591 | 19.70 |
| | 3 | 4.26 | 2.23 | 20.00 | 9.50 | 7.17 | 0.754 | 16.40 |
| | 4 | 4.41 | 2.26 | 20.00 | 9.97 | 6.80 | 0.682 | 20.70 |
| | 5 | 4.32 | 2.43 | 20.00 | 10.50 | 6.97 | 0.699 | 17.30 |
| | Average | 4.35 | 2.25 | 20.00 | 9.77 | 6.71 | 0.693 | 18.00 |

Figure 13: Tensile test raw data for Polymer C

CALCULATION OF THE STRAIN AND YOUNG'S MODULUS

Strain (or deformation) is defined as “the deformation of a solid due to stress” and is calculated by dividing the change in length of a solid over its original length: [7]

$$\epsilon = \frac{\Delta l}{l_0}$$

Using the calculation for “Test Number”: 1 of Polymer A as an example:

$$\frac{87.1 \text{ (mm)}}{20 \text{ (mm)}} = 4.36$$

The Young's Modulus of a solid is a measure of that solid's “stiffness” or “elasticity”; the higher the Young's Modulus the less the solid deforms under stress (stiffer). It is calculated by dividing the ultimate stress of an object by its strain: [7]

$$E = \frac{\sigma}{\epsilon}$$

Using the calculation for “Test Number”: 1 of Polymer A as an example:

$$\frac{0.487 \text{ (MPa)}}{4.36} = 0.11 \text{ (MPa)}$$

| Polymer Type | Test Number | Strain | Young's Modulus (Mpa) |
|--------------|-------------|--------|-----------------------|
| Polymer A | 1 | 4.36 | 0.11 |
| | 2 | 5.90 | 0.07 |
| | 3 | 6.65 | 0.07 |
| | 4 | 5.60 | 0.08 |
| | 5 | 4.84 | 0.09 |
| | Average | 5.47 | 0.09 |

Figure 14: Strain and young's modulus values for Polymer A

| Polymer Type | Test Number | Strain | Young's Modulus (Mpa) |
|--------------|-------------|--------|-----------------------|
| Polymer B | 1 | 0.69 | 0.99 |
| | 2 | 0.35 | 1.65 |
| | 3 | 0.31 | 1.38 |
| | 4 | 0.32 | 1.36 |
| | 5 | 0.39 | 1.26 |
| | Average | 0.41 | 1.33 |

Figure 15: Strain and young's modulus values for Polymer B

| Polymer Type | Test Number | Strain | Young's Modulus (Mpa) |
|--------------|-------------|--------|-----------------------|
| Polymer C | 1 | 0.80 | 0.93 |
| | 2 | 0.99 | 0.60 |
| | 3 | 0.82 | 0.92 |
| | 4 | 1.04 | 0.66 |
| | 5 | 0.87 | 0.81 |
| | Average | 0.90 | 0.78 |

Figure 16: Strain and young's modulus values for Polymer C

IR SPECTROSCOPY

POLYMER A

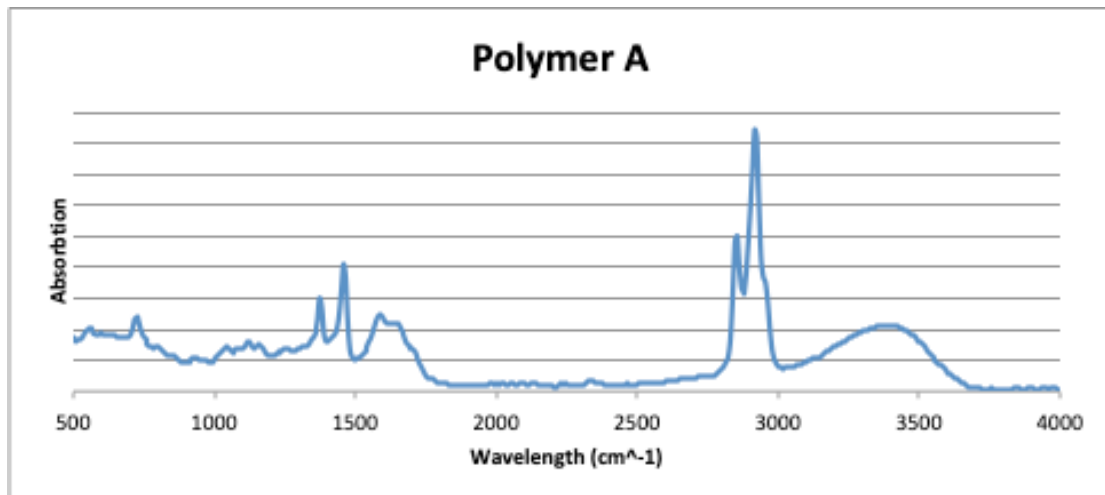


Figure 17: IR spectrum of Polymer A

POLYMER B

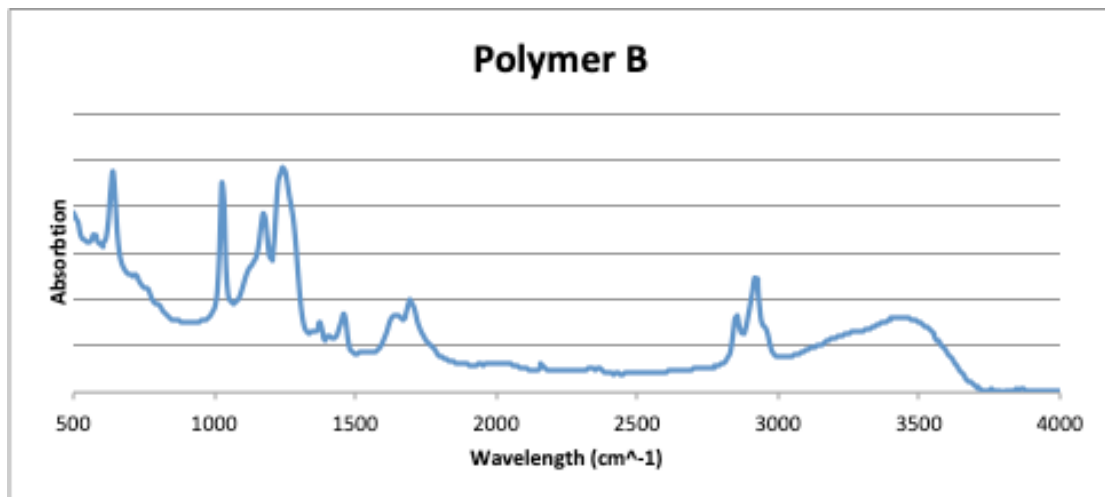


Figure 18: IR spectrum of Polymer B

POLYMER C

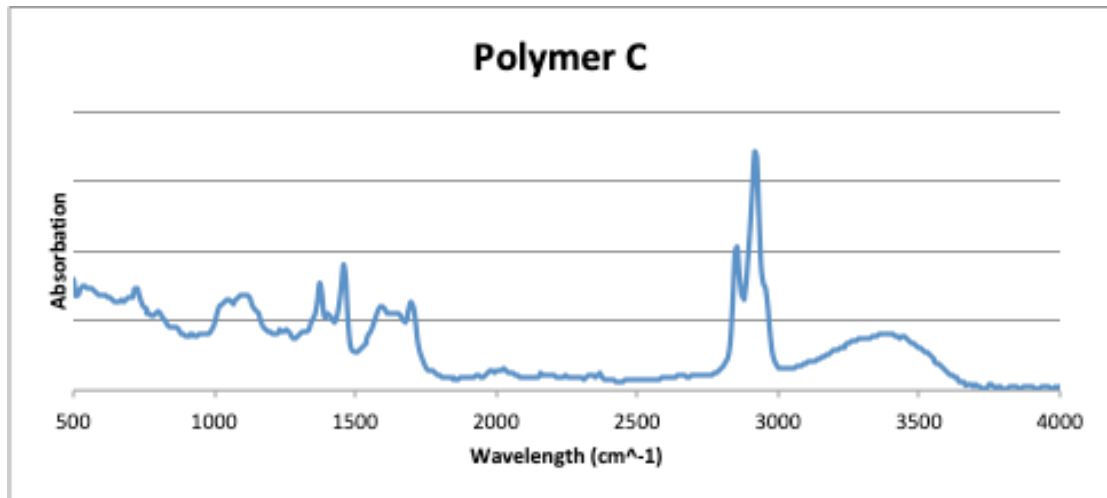


Figure 19: IR spectrum of Polymer C

SCANNING ELECTRON MICROSCOPY (SEM)

POLYMER B

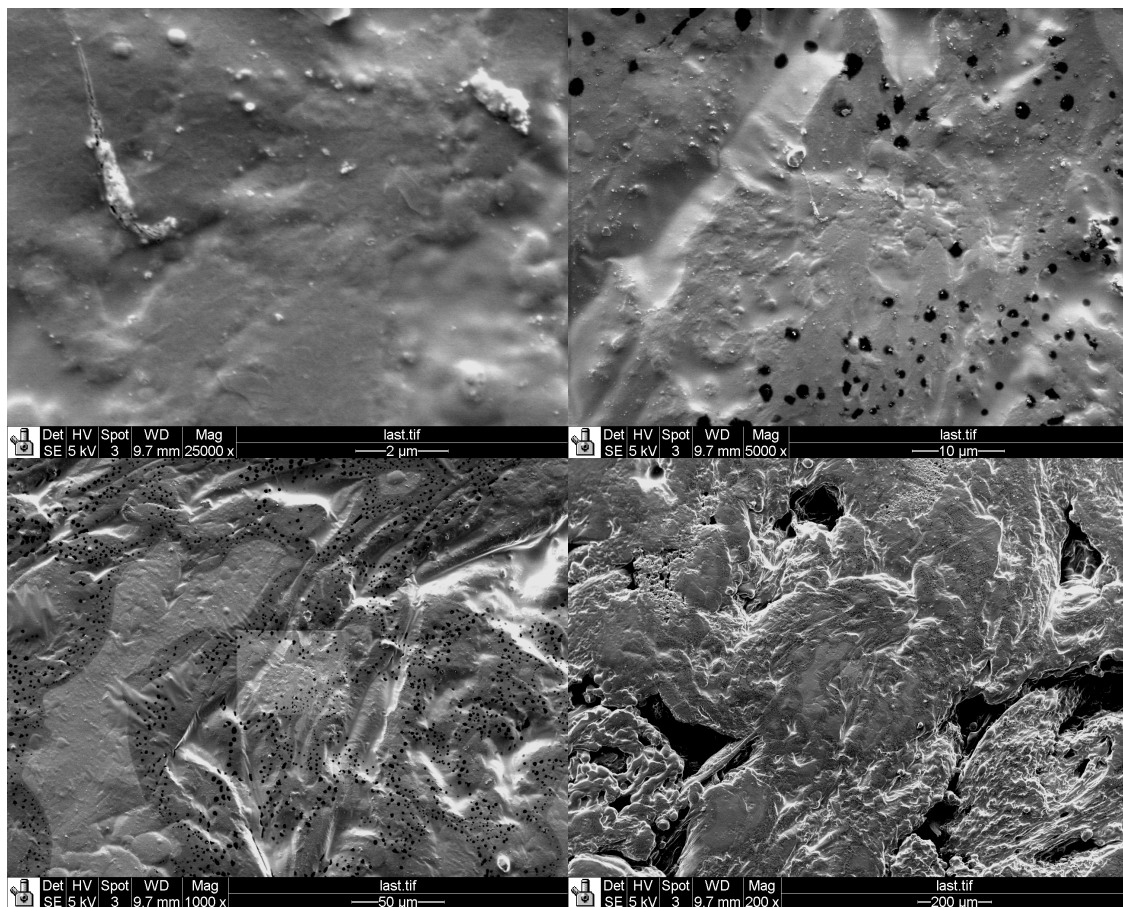


Figure 20: Scanning Electron Microscopy images for Polymer B

POLYMER C

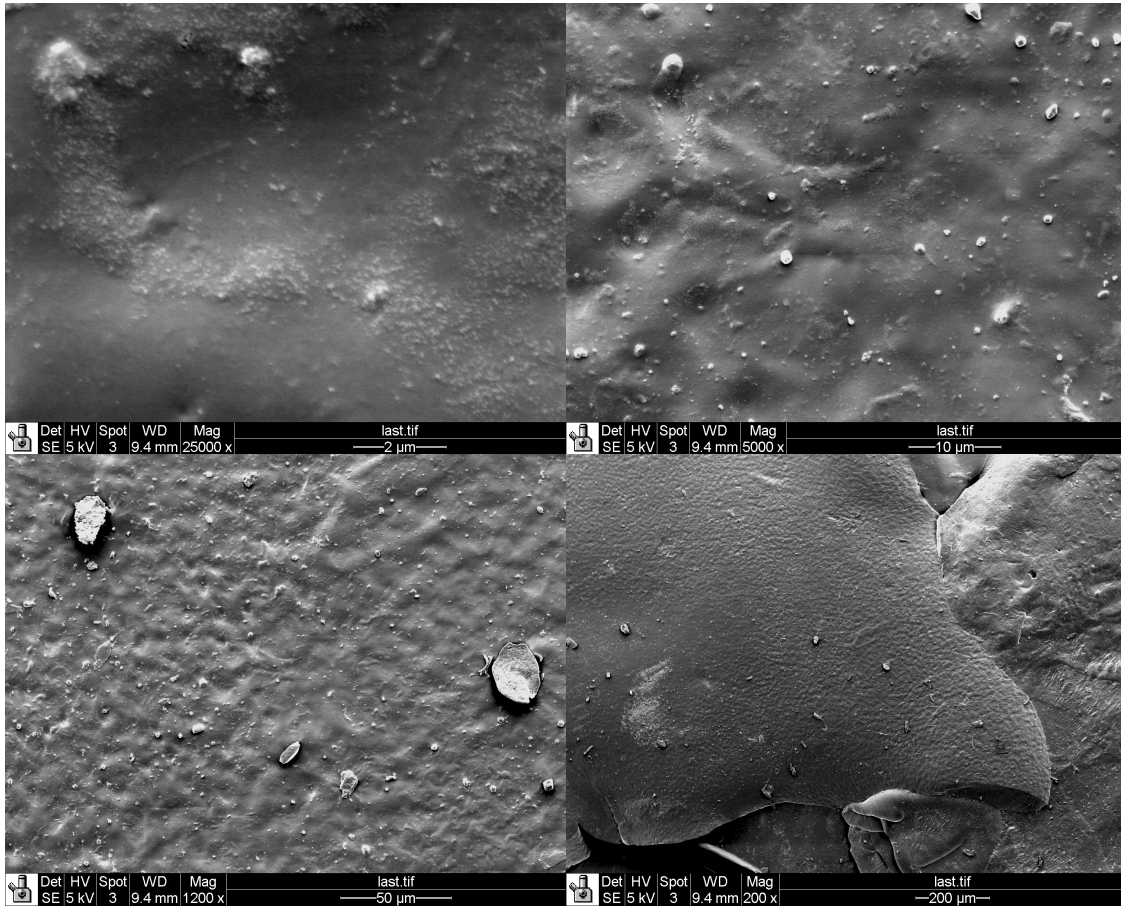
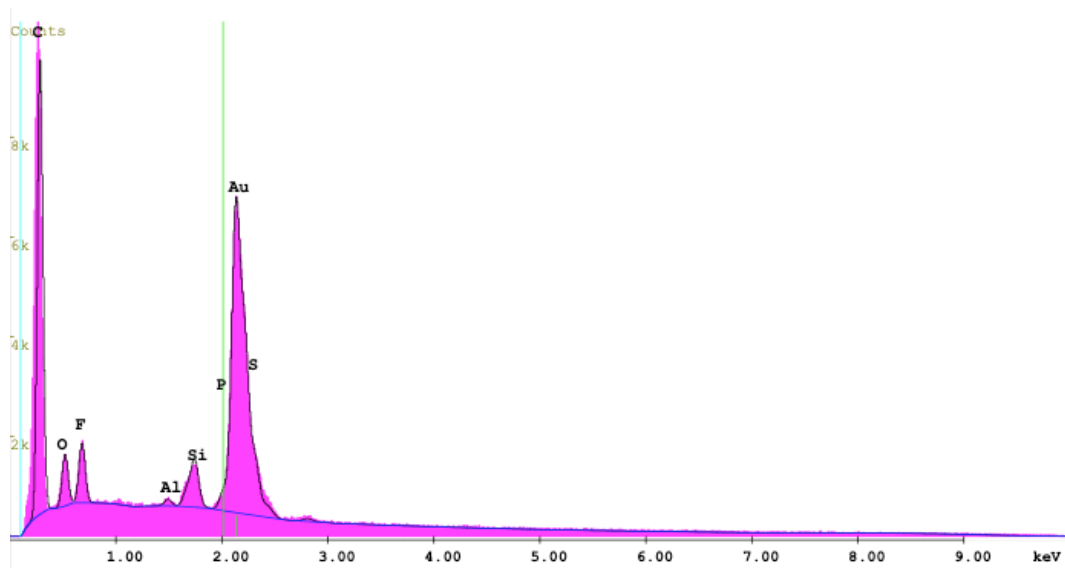


Figure 21: Scanning Electron Microscopy images for Polymer C

ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (EDS)

POLYMER B

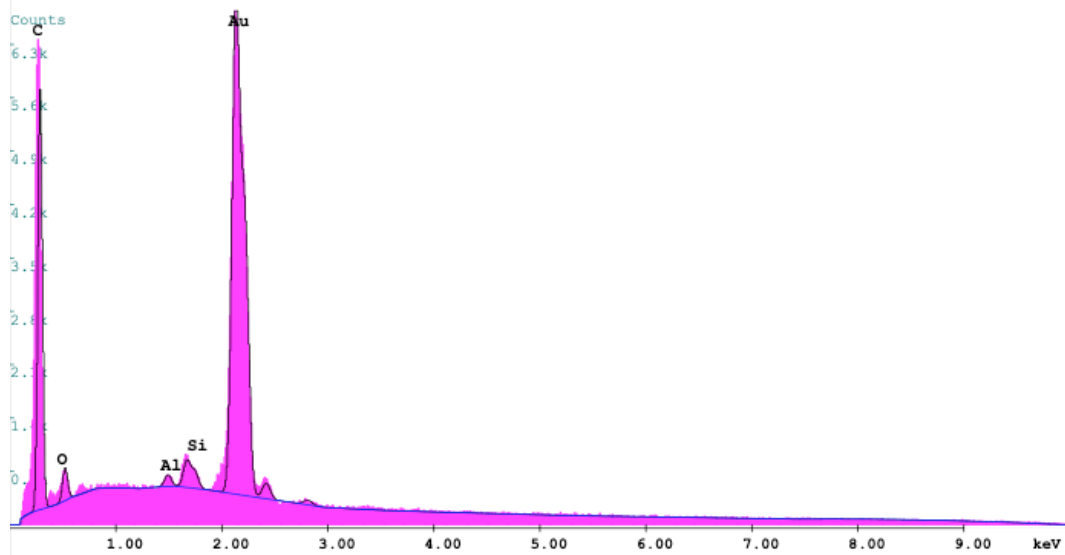


| Element | Wt % | At % | K-Ratio | Z | A | F |
|---------|--------|--------|---------|--------|--------|--------|
| C K | 73.90 | 84.12 | 0.3930 | 1.0182 | 0.5222 | 1.0001 |
| O K | 6.74 | 5.76 | 0.0275 | 0.9956 | 0.4095 | 1.0002 |
| F K | 5.38 | 3.87 | 0.0286 | 0.9333 | 0.5691 | 1.0001 |
| SiK | 4.19 | 2.04 | 0.0386 | 0.9357 | 0.9796 | 1.0052 |
| P K | 2.50 | 1.10 | 0.0223 | 0.9027 | 0.9828 | 1.0063 |
| S K | 7.29 | 3.11 | 0.0666 | 0.9230 | 0.9888 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

| Element | Net Inte. | Bkgd Inte. | Inte. Error | P/B |
|---------|-----------|------------|-------------|-------|
| C K | 825.64 | 52.30 | 0.46 | 15.79 |
| O K | 104.93 | 82.64 | 1.95 | 1.27 |
| F K | 124.67 | 90.78 | 1.75 | 1.37 |
| SiK | 111.89 | 88.77 | 1.90 | 1.26 |
| P K | 54.65 | 86.67 | 3.45 | 0.63 |
| S K | 144.84 | 70.72 | 1.46 | 2.05 |

Figure 22: EDS of Polymer B

POLYMER C



| Element | Wt % | At % | K-Ratio | Z | A | F |
|---------|--------|--------|---------|--------|--------|--------|
| C K | 86.81 | 90.93 | 0.7271 | 1.0049 | 0.8335 | 1.0001 |
| O K | 9.34 | 7.34 | 0.0369 | 0.9827 | 0.4021 | 1.0000 |
| SiK | 3.85 | 1.73 | 0.0350 | 0.9230 | 0.9843 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

| Element | Net Inte. | Bkgd Inte. | Inte. Error | P/B |
|---------|-----------|------------|-------------|-------|
| C K | 224.44 | 9.90 | 0.70 | 22.67 |
| O K | 20.69 | 18.96 | 3.70 | 1.09 |
| SiK | 14.91 | 42.98 | 6.74 | 0.35 |

Figure 23: EDS of Polymer C

DISCUSSION

TENSILE TESTS

The average young's modulus of Polymer A (0.09 MPa) is the lowest of the three, indicating that it is the most elastic, or least stiff. This is logical as Polymer B and C are cross-linked whereas Polymer A is not; crosslinking a polymer leads to a stiffer overall material, as the structure is held together by more bonds, relative to Polymer A.

Polymer C has an average young's modulus of 0.78 MPa; lower than that of B but higher than that of A. Out of the two cross-linked Polymers, B is stiffer (more rigid) than C, with the young's modulus of Polymer B (1.33) being just less than twice that of Polymer C (0.78). This could be seen as an indication that crosslinking is more effective when utilizing the synthesis route to obtain Polymer B.

Polymer C exhibited a higher average ultimate stress (0.693 MPa) relative to Polymer B (0.522 MPa), with both demonstrating higher average ultimate stresses than Polymer A (0.461 MPa). This indicates that crosslinking has indeed strengthened the polymers, with Polymer C being the strongest. Polymer B possesses a lower average young's modulus, relative to Polymer C; Polymer C is stronger than B, however Polymer C less stiff, or more elastic, relative to Polymer B. Perhaps the degree of crosslinking combined with differences in the shapes and sizes of the filler molecules are responsible for these results.

IR SPECTROSCOPY

POLYMER A

A large peak can be observed between 2900 and 3000 cm^{-1} along with a smaller one ending at about 2900 cm^{-1} , both of which indicating the presence of alkyl, methylene C-H bonds. A weak broad peak can be observed between 1500 and 1800 cm^{-1} , which indicates the presence of C=O anhydride bonds. Two peaks are present just below 1500 cm^{-1} , the one nearest to 1500 cm^{-1} indicating the possible presence of aromatic C=C bonds, while the second weaker peak may indicate the presence of alkyl, methyl C-H bonds. The broad band from 3000 cm^{-1} to 3600 cm^{-1} may indicate the presence of secondary amine N-C bonds, however it may also indicate the presence of alcohol O-H bonds present from ethanol used to clean the brabender moulds. [8]

POLYMER B

The same peaks are present as in the IR spectrum of Polymer A. (Refer to "Discussion: IR Spectroscopy: Polymer A")

Three new distinct peaks can now be observed between: 600 cm^{-1} to 700 cm^{-1} , 1000 cm^{-1} to 1100 cm^{-1} , and 1100 cm^{-1} to 1300 cm^{-1} . The peak from 1100 cm^{-1} to 1300 cm^{-1} is indicative of Si-O network bonds [9], which confirm the crosslinking of Polymer A was successful. The latter two peaks cannot be accounted for without considering the contamination of the polymer; the peak from 1000 cm^{-1} to 1100 cm^{-1} strongly suggests the presence of C-F bonds, likely originating from the trifluoromethylsulfonic acid used during the hydrolysis/condensation of the silica nanoparticles, while the peak from 600 cm^{-1} to 700 cm^{-1} could similarly be explained by a C-H alkene vinyl group, resulting from the acid mutilating the newly cross-linked polymer. [8]

POLYMER C

The same peaks are present as in the IR spectrum of Polymer A. (Refer to "Discussion: IR Spectroscopy: Polymer A")

One new peak is present relative to the spectrum of Polymer A (1100 cm^{-1} to 1300 cm^{-1}).^[9] This is indicative to Si-O network bonds indicating the successful crosslinking of Polymer A and the formation of Polymer C. The lack of other new peaks (relative to the spectrum of Polymer A) suggests that Polymer C does not suffer from the impurities present in Polymer B.

SCANNING ELECTRON MICROSCOPY (SEM)

POLYMER B

At the $200\mu\text{m}$ range one can observe the texture of the polymer resembling a melted, coagulated quality, in stark contrast to Polymer C. This difference in texture is likely one of the reasons for the lower average ultimate stress of Polymer B relative to Polymer C.

At the range one can observe small white spots littering the image non-uniformly and random. It is likely these are the silica nanoparticles, as they should be around 100nm ^{[10][11]} in diameter. They appear to be larger relative to those in observed in the SEM of Polymer C. Again this difference in size is, likely, one of reasons for the disparity between the average ultimate stresses of Polymers B and C.

POLYMER C

At the $200\mu\text{m}$ range the structure of Polymer C appears layered and segmented much unlike Polymer B.

When the SEM was performed $2\mu\text{m}$ (2000nm) small white spots could be observed on the polymer, however unlike Polymer B these appear smaller and are only present in specific areas; they are uniform and non-random. There also appears to be more of them in the SEM image of Polymer C relative to the SEM image of Polymer B, however this could differ in other areas of both polymers. Perhaps the grouping of the silica nanoparticles provides Polymer C with greater structure, thereby explaining its higher average ultimate stress, relative to Polymer B.

ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (EDS)

POLYMER B

Peaks for fluorine and sulphur were observed confirming that the peaks observed in the IR spectrum of Polymer B, were due to bonds between these elements. Again it is likely that this contamination is, due to the trifluoromethylsulfonic acid added to initiate the hydrolysis/condensation of ethoxysilyl ends of the maleimide.

The phosphorous peak should be impossible. This element was never used throughout any of the experiments. The most likely explanation is that, due to multiple experiments that were performed in tandem, within the same fume hood, alongside this experiment, cross contamination occurred.

Finally, the presence of the: carbon, oxygen and silicon, peaks reaffirms, along with the IR spectrum of Polymer B, that crosslinking was successful.

POLYMER C

The phosphorous, sulphur and fluorine peaks of Polymer B's EDS are not present in the EDS of Polymer C, while the silicon and oxygen peaks are. Along with the IR spectrum of Polymer B this strongly indicates that the crosslinking Polymer C was achieved without the contamination present in Polymer B.

CONCLUSION

Crosslinking of both polymers was successful, however Polymer B contained contamination, likely, from the trimethylfluorosulfonic acid, used to initiate the hydrolysis/condensation of the ethoxysilyl ends of the 3-(1-triethoxysilylpropyl)maleimide, and another unknown source.

Polymer C constantly exhibited a higher ultimate stress, relative to Polymer B, while Polymer B demonstrated a higher young's modulus, relative to Polymer C. Both polymers B and C consistently possessed higher ultimate stresses and young's moduli, relative to Polymer A.

The SEMs at the 200 μ m range presented Polymer B with a coagulated texture, whereas Polymer C appeared more encrusted. At the 2 μ m range of the SEMs small white dots could be observed on the surface of both polymers, around 100 nm in diameter. It is highly likely these are the silica nanoparticles. The dots in the SEM of Polymer C appear smaller and more uniform, in both size and location, relative to those observed in Polymer B.

Performing the Diels-Alder before the hydrolysis/condensation of ethoxysilyl ends of the 3-(1-triethoxysilylpropyl)maleimide, appears to yield a polymer with superior tensile properties, relative to the polymer formed when the hydrolysis/condensation of ethoxysilyl ends of the 3-(1-triethoxysilylpropyl)maleimide occurs before the Diels-Alder reaction.

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