Compatibilization of PP Nanocomposites: High Processing Temperature with EFM

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PNC-Tech Meeting [Proceedings], 2005-09-28

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IMI-ITRI Joint Program

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Objectives

- Evaluation of effect of clay type and intercalation coverages on the dispersion and mechanical properties of PP based nanocomposites.
- Evaluation of compounding conditions on the dispersion and mechanical properties of PP-based nanocomposites

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Materials and codes

Material (code)	Intercalant types and coverage levels (%)
PP	Profax 1274, Basell
PB3150	Polybond 3150, Crompton
UCL01 (quaternary amine)	Trimethylstearyammonium Chloride, 100
UCL03 (ternary amine)	N, N-dimethyl-n-octadecylamine, 100
UCL04 (ternary amine)	N, N-dimethyl-n-octadecylamine, 60
UCL05 (primary amine)	Octadecylamine, 100
UCL06 (primary amine)	Octadecylamine, 60

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Sample Designation --- TSE

Designation	Loading and organoclay type	Loading and compatibilizer type
PP		
PPMA		4 wt% of Polybond 3150
TNQ100B	2 wt% of UCL01	4 wt% of Polybond 3150
TNT100	2 wt% of UCL03	4 wt% of Polybond 3150
TNT60	2 wt% of UCL04	4 wt% of Polybond 3150
TNP100	2 wt% of UCL05	4 wt% of Polybond 3150
TNP60	2 wt% of UCL06	4 wt% of Polybond 3150

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Sample Designation --- SSE and SSE + EFM

Designation	Loading and organoclay type	Loading and compatibilizer type
SNQ100B	2 wt% of UCL01	4 wt% of Polybond 3150
SNQ100BE	2 wt% of UCL01	4 wt% of Polybond 3150
SNT100	2 wt% of UCL03	4 wt% of Polybond 3150
SNT100E	2 wt% of UCL03	4 wt% of Polybond 3150
SNT60	2 wt% of UCL04	4 wt% of Polybond 3150
SNT60E	2 wt% of UCL04	4 wt% of Polybond 3150
SNP100	2 wt% of UCL05	4 wt% of Polybond 3150
SNP100E	2 wt% of UCL05	4 wt% of Polybond 3150
SNP60	2 wt% of UCL06	4 wt% of Polybond 3150
SNP60E	2 wt% of UCL06	4 wt% of Polybond 3150

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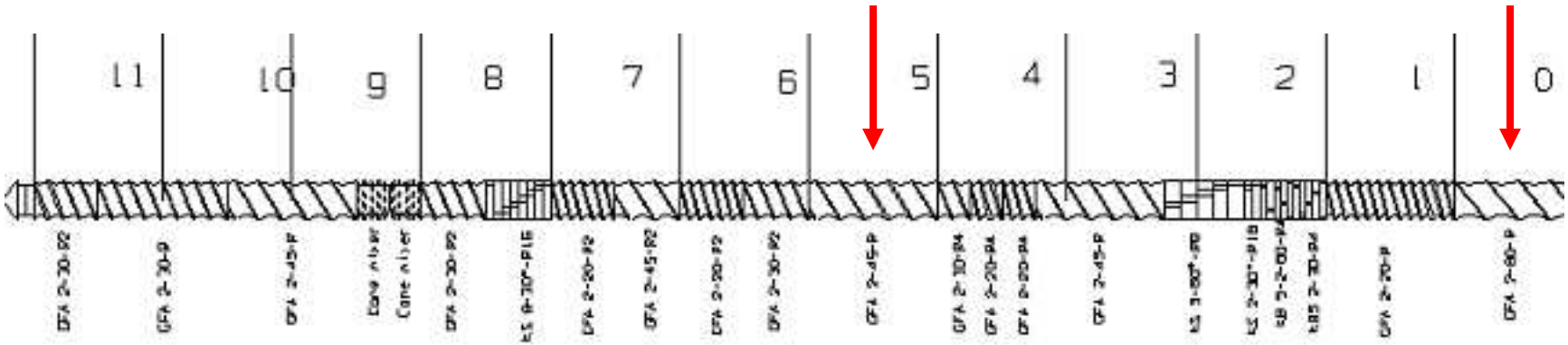
Processing Condition --- TSE



TSE: Leistritz-34mm CORI, L/D = 40 with screw configuration shown above (*N* = 200 rpm, *T* = 190°C, *Q* =5 kg/h), under a blanket of dry nitrogen.

Feeding: Zone 0: PP + compatibilizers;

Zone 5: side feeder for orgaoclay

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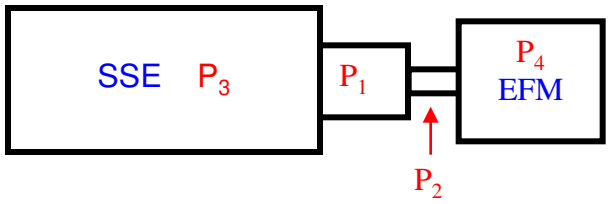
Processing Conditions ---SSE or SSE +EFM



Residence time: 1163 s (~20 min)

No	Samples	Device(s)	EFM Gap (μm)	Temp. (°C)	RPM	Flow Rate (kg/h)	Operation Pressure (Psi) P1	Operation Pressure (Psi) P2	Operation Pressure (Psi) P3	Operation Pressure (Psi) P4	Organocla y (wt%)
1	PP	SSE	---	190	5	5	550	-	-	-	2
2	PP+PPMA	SSE	---	190	6	5	579	-	-	-	2
3	SSENPQ100B	SSE	---	190	6	5	525	-	-	-	2
4	SSENPT100	SSE	---	190	10	5	1116	-	-	-	2
5	SSENPT60	SSE	---	190	7	5	569	-	-	-	2
6	SSENPP100	SSE	---	190	9	5	1091	-	-	-	2
7	SSENPP60	SSE	---	190	10	5	1128	-	-	-	2
8	SSENPQ100BE	SSE+EFM-3	30	190	17	5	2162-2642	2454-2466	3000-3008	2297-2310	2
9	SSENPT100E	SSE+EFM-3	30	190	26	5	3225-3257	3467-3533	3970-4033	3260-3333	2
10	SSENPT60E	SSE+EFM-3	30	190	36	5	3250-3359	4048-4065	5247-5383	3862-3887	2
11	SSENPP100E	SSE+EFM-3	30	190	41	5	3704-3708	4255-4265	5300-5310	4119-4131	2
12	SSENPP60E	SSE+EFM-3	30	190	47	5	3979-4045	4939-5066	6282-6445	4780-4880	2

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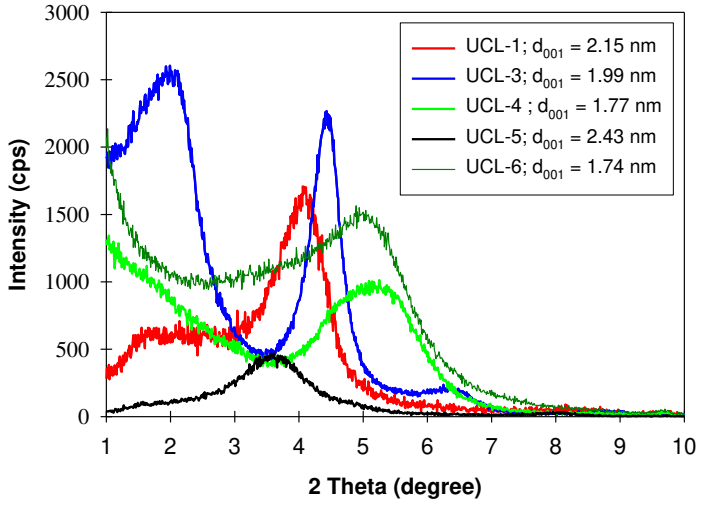
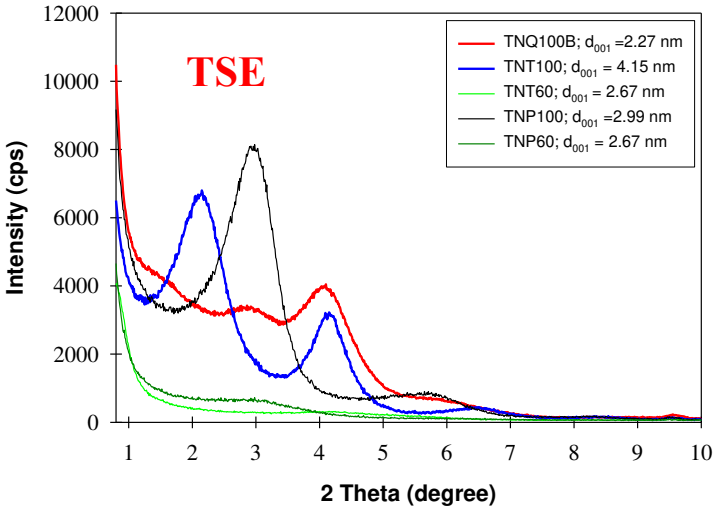
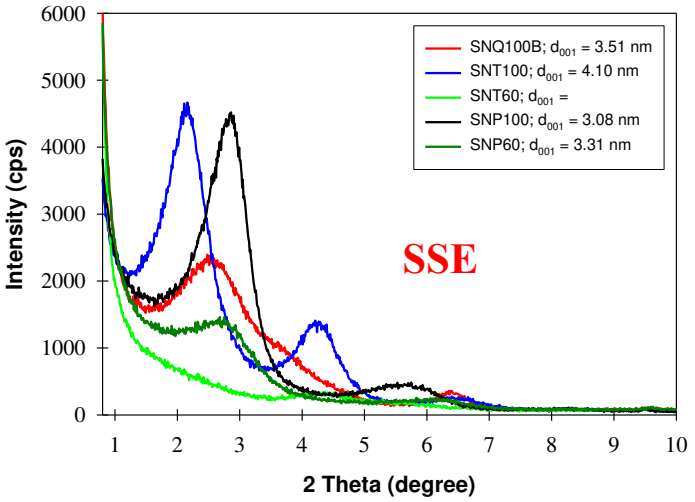
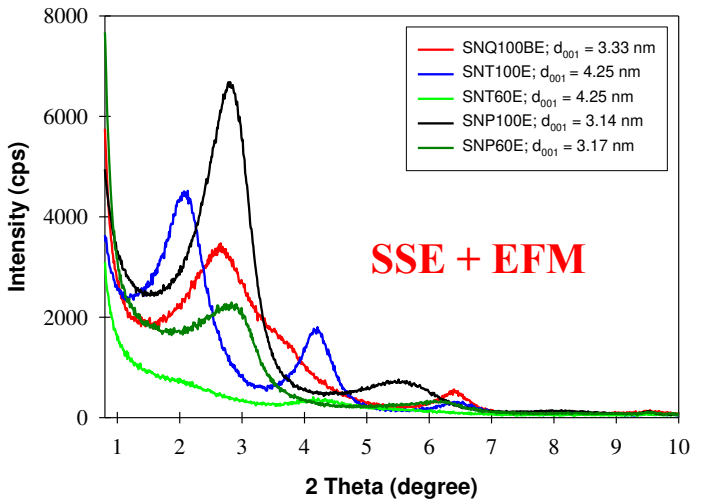
Dispersion -XRD









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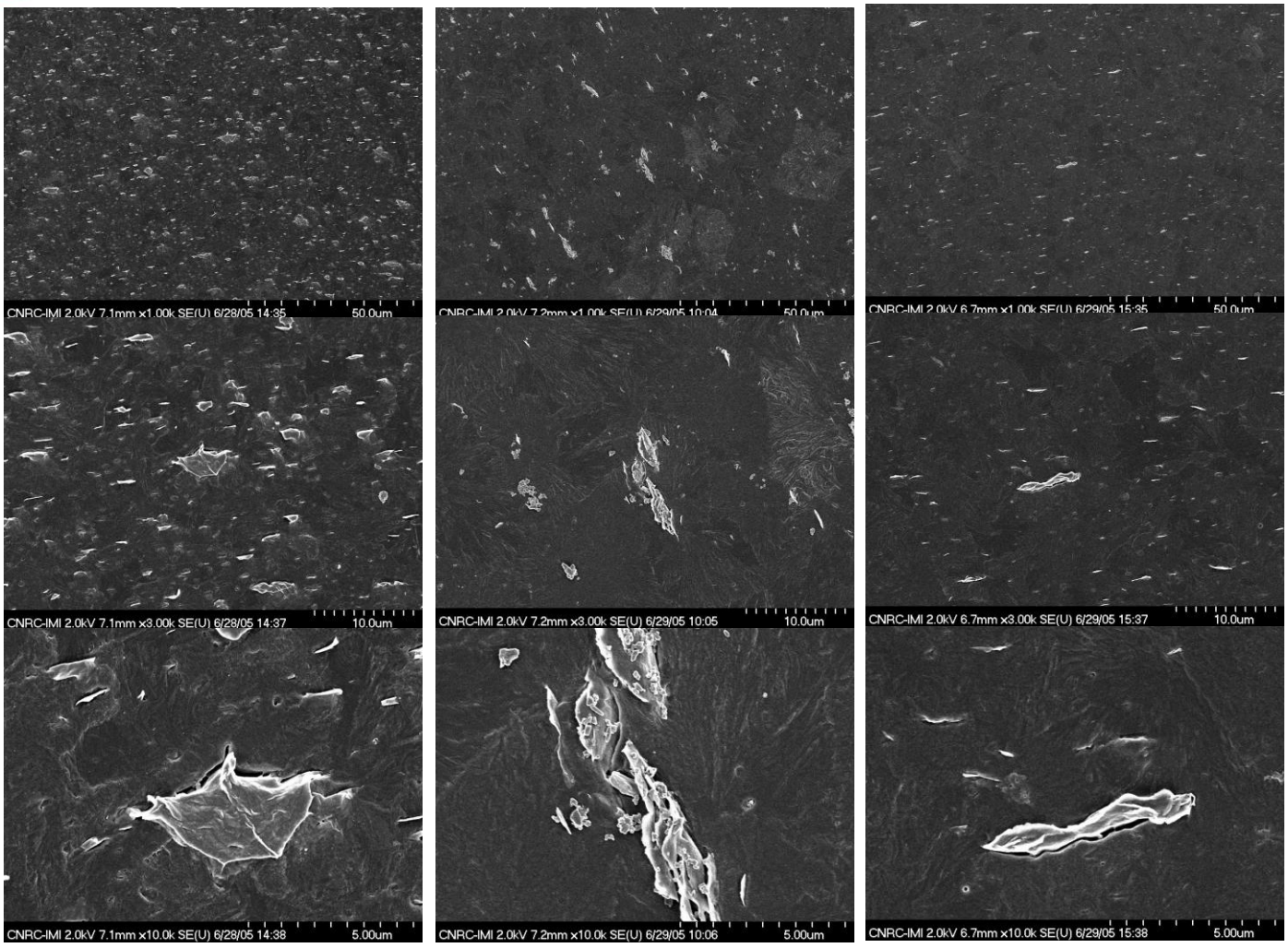
Dispersion --- FEGSEM (1)

Magnification



TNQ100B **SNQ100B** **SNQ100BE**

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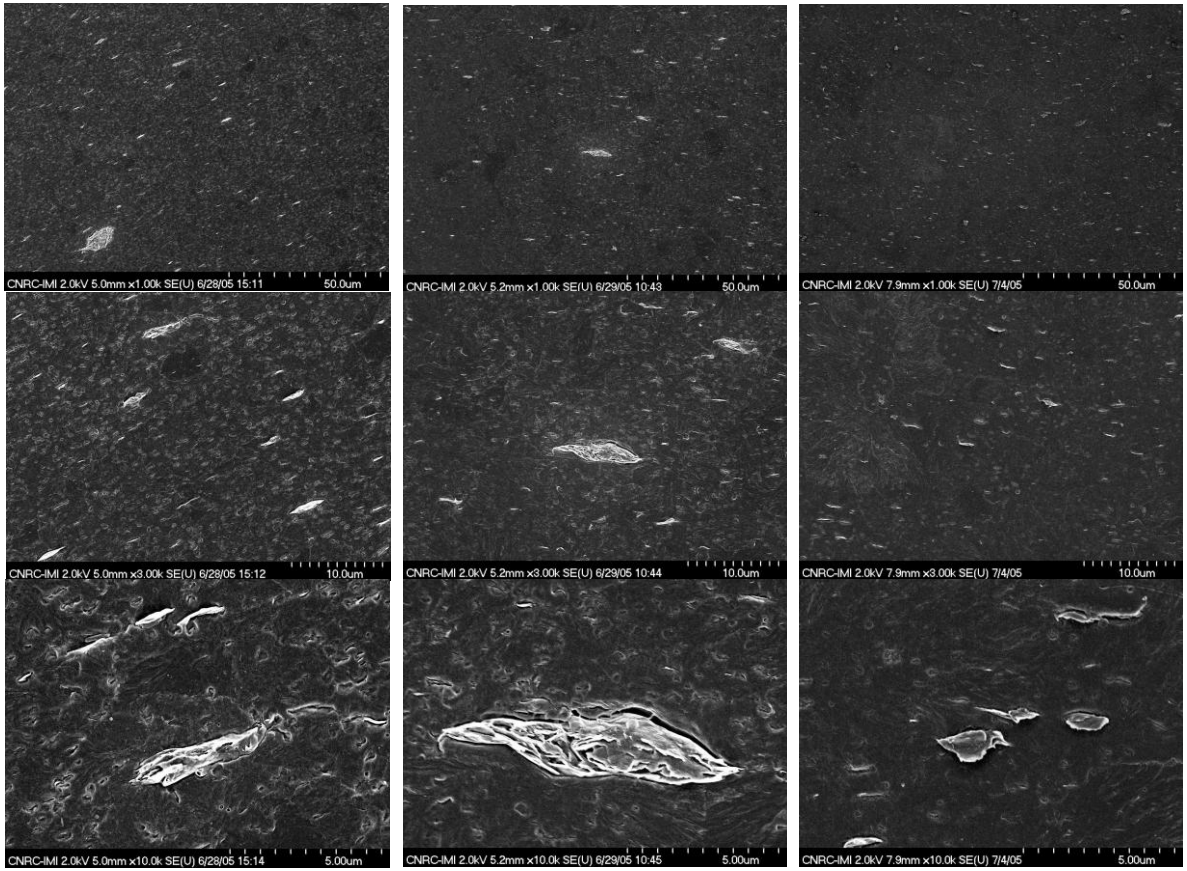
Dispersion --- FEGSEM (2)

Magnification



TNT100 **SNT100** **SNT100E**

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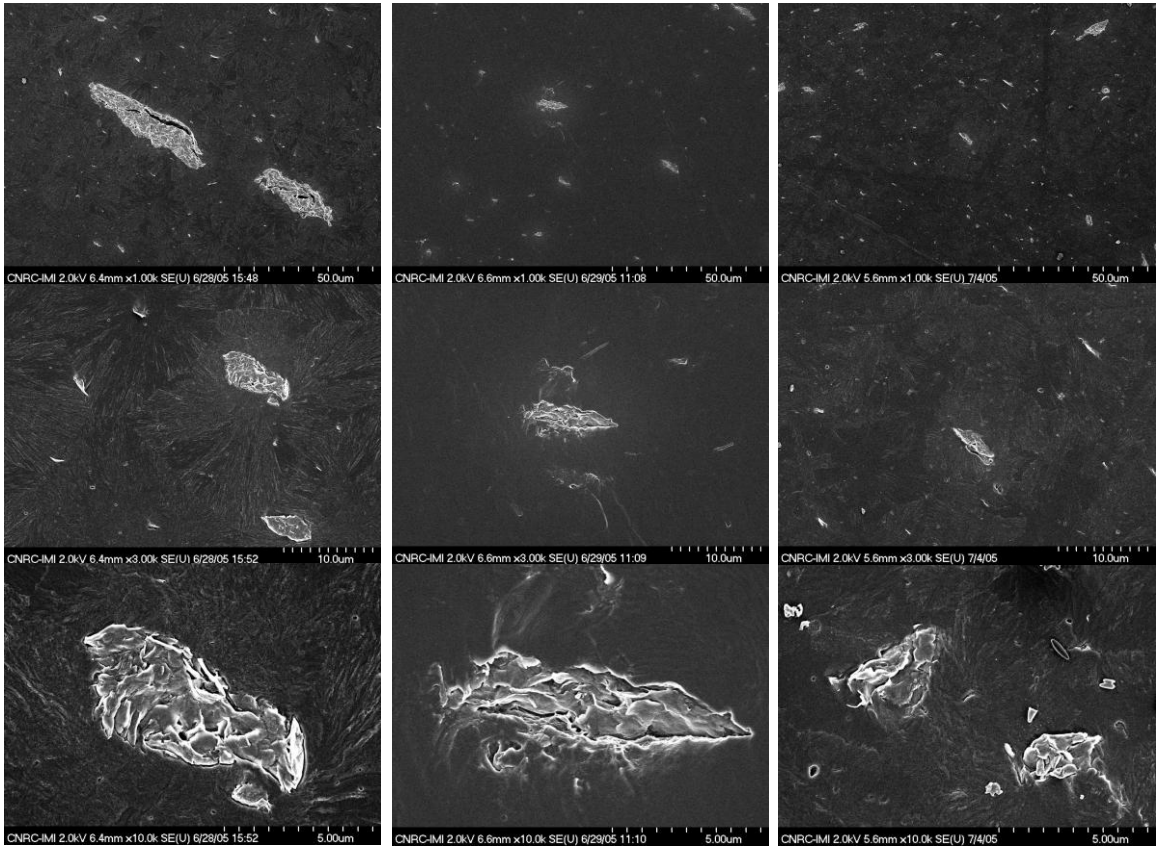
Dispersion --- FEGSEM (3)

Magnification



TNT60 SNT60 SNT60E

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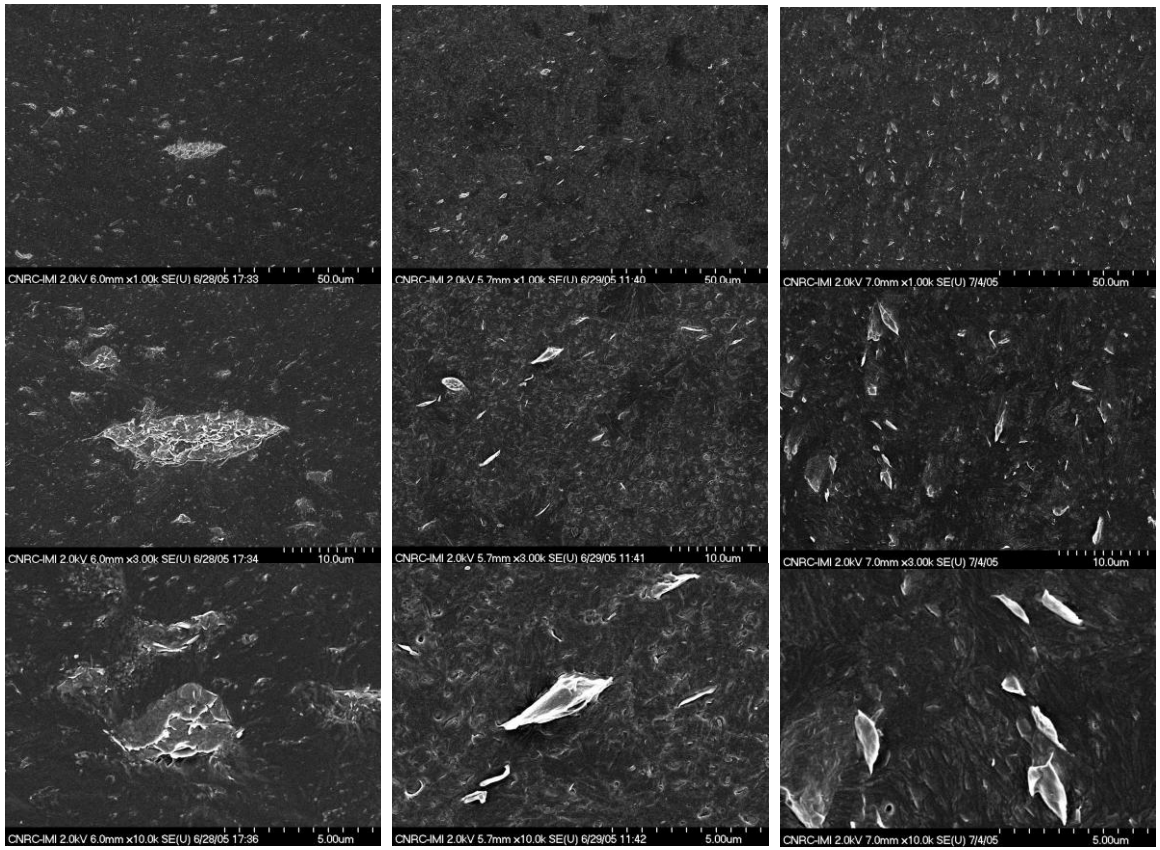
Dispersion --- FEGSEM (4)

Magnification



TNP100 **SNP100** **SNP100E**

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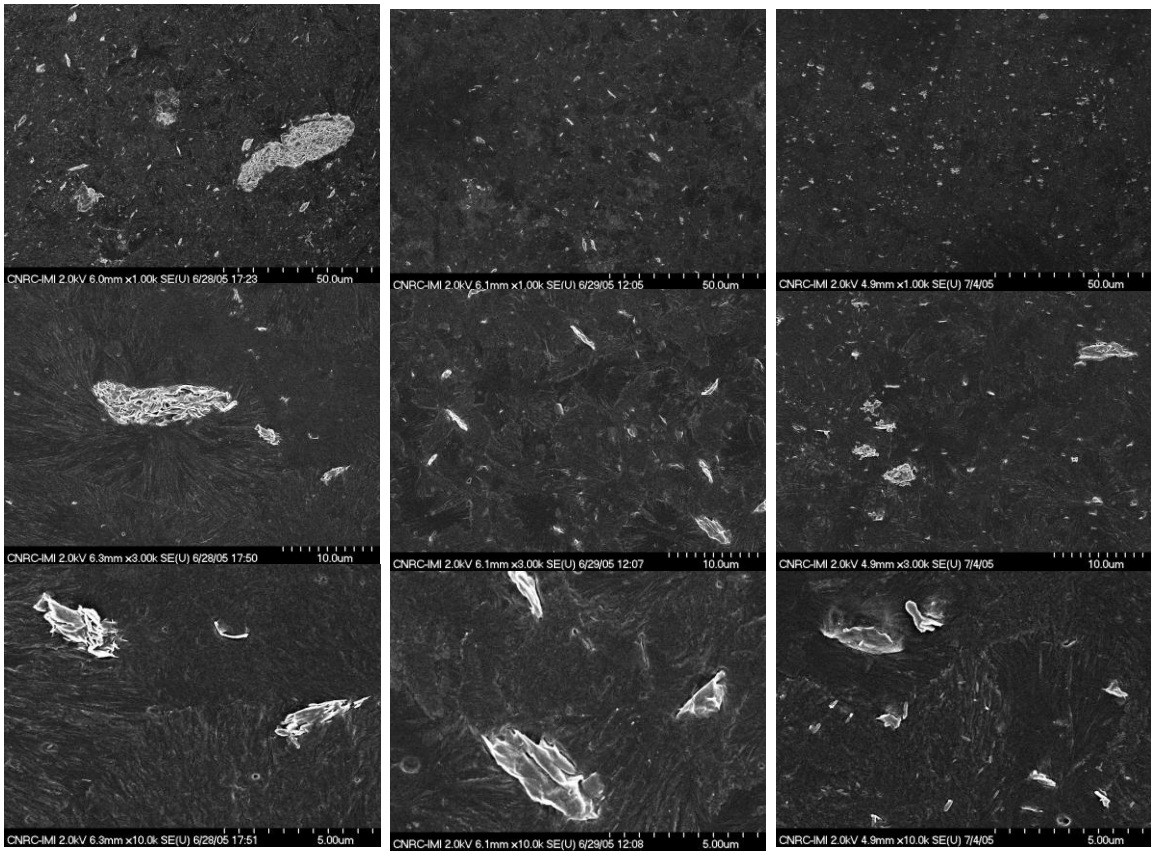
Dispersion --- FEGSEM (5)

Magnification



TNP60 **SNP60** **SNP60E**

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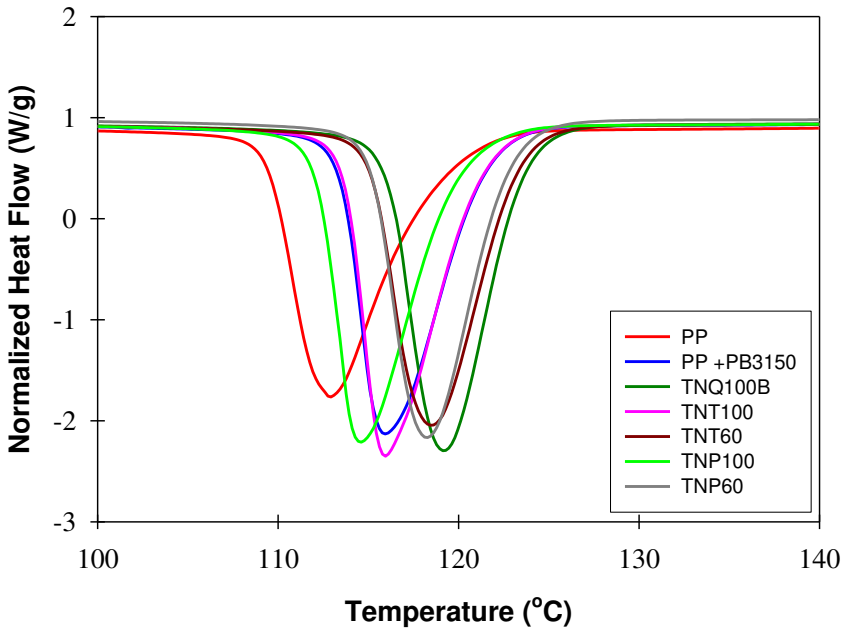
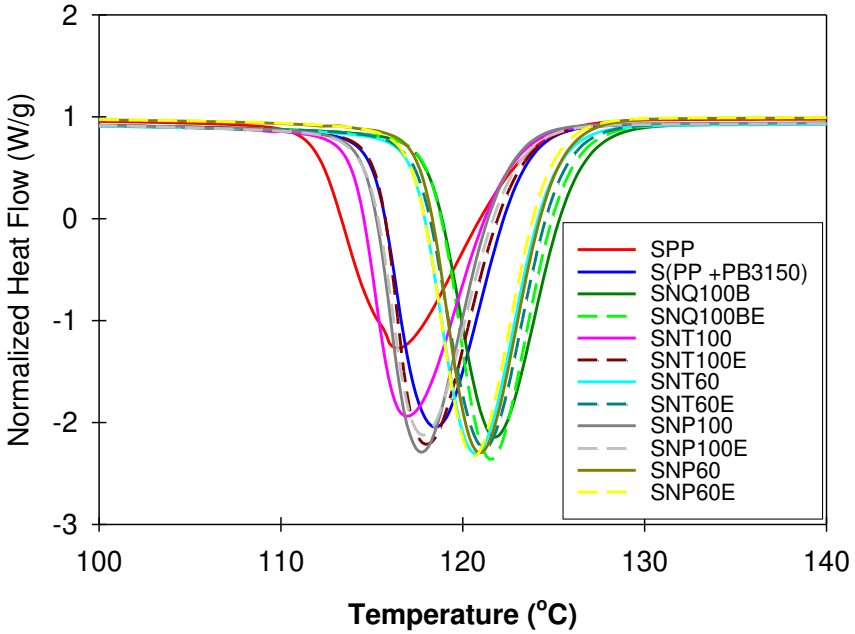
DSC Thermogram

TSE



SSE or SSE + EFM



DSC Measurement --- TSE

Samples	T_{onset}	T_{peak}	Crystallinity (%)
PP	118.7	112.9	47.0 (0.2)
PP+PB3150	121.4	116.2	46.7 (0.3)
TNQ100B	123.7	119.3	45.6 (0.4)
TNT100	120.2	115.9	45.8 (0.2)
TNT60	123.5	118.5	46.4 (0.5)
TNP100	120.1	114.6	46.1 (0.3)
TNP60	122.7	118.0	45.6 (0.3)

- Addition of organoclay accelerates the crystallization rate due to the nucleation effect.
- No significant variation of crystallinity between the matrix and nanocomposites

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DSC Measurement --- SSE

Samples	T_{onset}	T_{peak}	Crystallinity (%)
PP	122.6	115.9	46.7 (0.2)
PP+PB3150	123.4	118.5	46.1 (0.3)
SNQ100B	126.2	121.7	45.1 (0.2)
SNQ100BE	125.8	121.7	45.5 (0.3)
SNT100	122.6	117.0	45.4 (0.2)
SNT100E	122.7	117.7	44.9 (0.2)
SNT60	125.0	120.9	45.2 (0.1)
SNT60E	125.7	121.5	45.8 (0.1)
SNP100	122.4	117.7	44.9 (0.2)
SNP100E	122.6	117.9	45.3 (0.1)
SNP60	125.1	120.8	46.3 (0.1)
SNP60E	124.7	120.6	45.6 (0.2)

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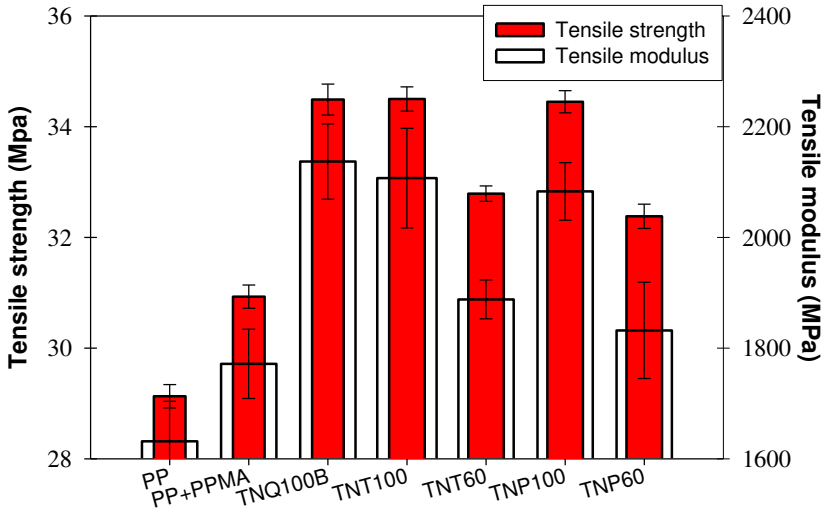
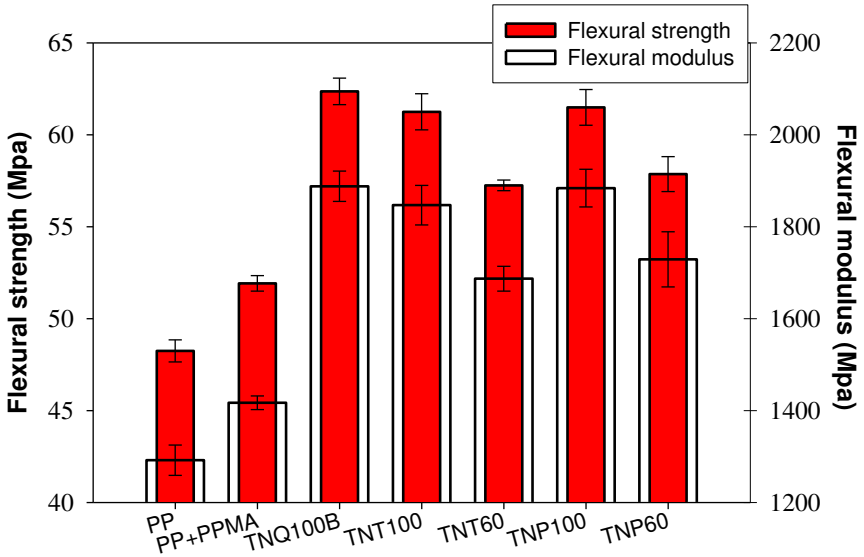
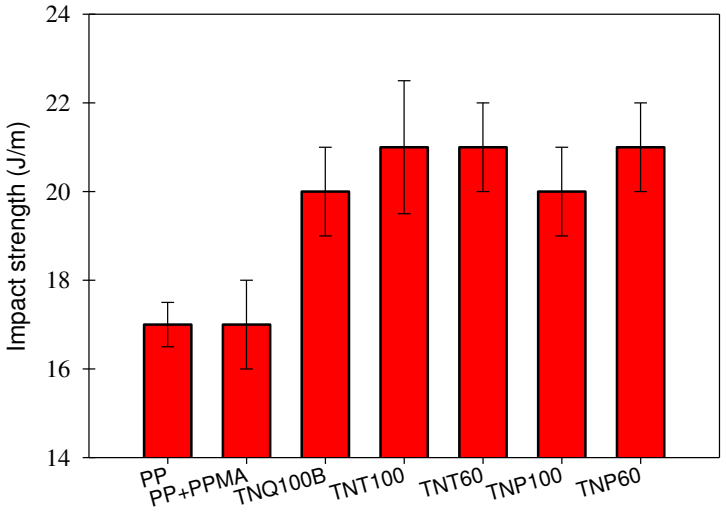
Mechanical Properties -- TSE

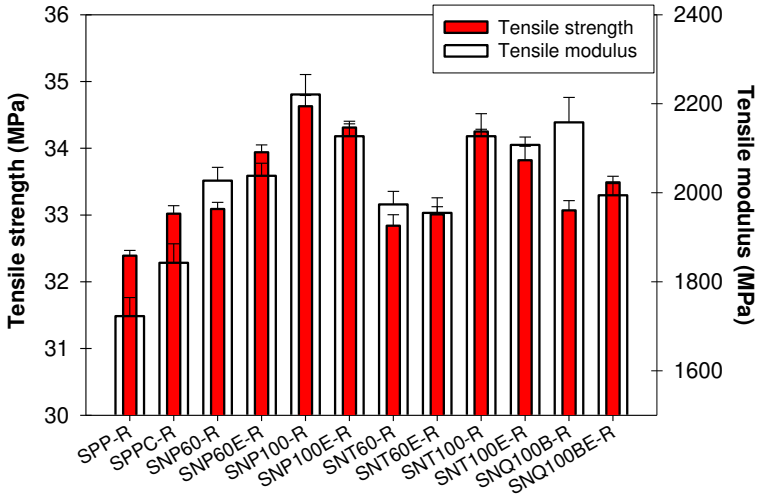


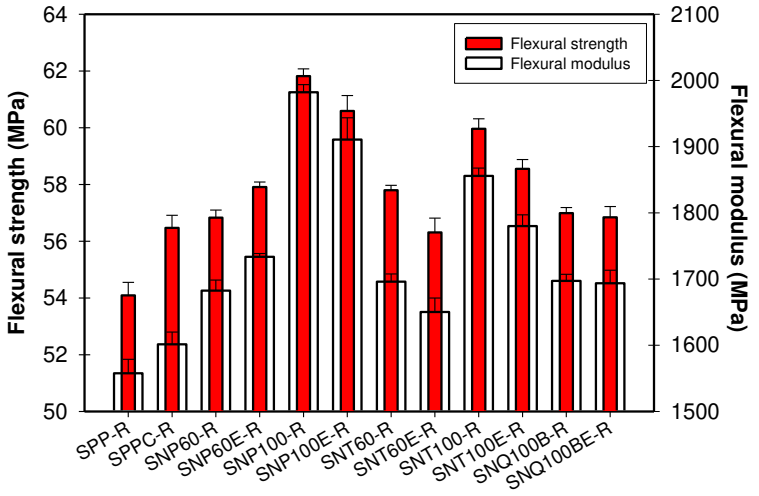


- TNQ100, TNT100 and TNP100 display significant improvement on tensile properties.
- Similar case appears on flexural properties.
- little difference is found on impact strength for PNC, no mater what type of clay and what intercalant coverages.

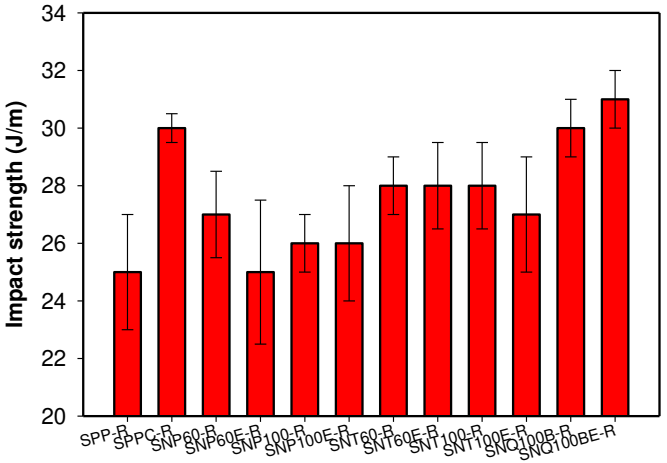


Mechanical Properties
--- by SSE and SSE+EFM





- SNP100, SNT100 and SNQ100 display significant improvement on tensile modulus.
- Similar case for SNP100 and SNT100 on flexural properties.
- No significant improvement was observed for PNC on impact strength.



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Summary

- Comparing the samples prepared from TSE, SSE and SSE+EFM, it seems that the residence time played role during the melt intercalation. In the case of dispersion the compounding devices are in the order of: SSE+EFM, SSE and TSE.
- Big aggregates were observed for the samples prepared either by TSE or SSE. The sample prepared by SSE+EFM displayed more uniform dispersion --- effect of EFM.
- The effect of EFM was obvious for the microdispersion but not for the further intercalation. The high operation pressure on the one hand improved delamination of clay particles and on the other hand caused the degradation of intercalant thus resulting the reduction of d spacing of organoclay.
- Crystallization rate was increased by the addition of compatibilizer and organoclay in all above compounding conditions but not the case for the samples containing UCL03 and UCL05. No significant crystallinity variation was found between the matrix and PNCs. (Cont…….)

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Summary (cont.)

- Samples with higher intercalant coverages demonstrated better tensile and flexural properties in all compounding conditions. The impact strength of PNC prepared by TSE was better than matrix, but no significant difference was observed in terms of effect of clay types and intercalant coverages.
- In order to get better dispersion of organoclay in PP matrix and enhancement of mechanical properties of PNC the masterbatch step during melt processing is indispensable.