



Improving the Vicat Softening Point of Poly (Vinyl Chloride) mixtures through blending with different polymers and inorganic fillers



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Abstract

To improve the Vicat Softening Point of PVC compounds, blending technique was used to blend PVC with other polymers such as Acrylonitrile butadiene styrene (ABS), Acrylonitrile styrene acrylate (ASA) and Poly (methyl methacrylate) (PMMA) at different ratios (5, 10 and 20 %), then comparing the mechanical and thermal properties obtained with commercial heat modifier polymer Alpha Methyl Styrene-Acrylonitrile copolymer (AMSAN) The blended PVC mixtures were studied to find that, the best polymer that can be used to improve the Vicat Softening point. It was the blend of PVC with PMMA. Also, the effect of adding inorganic fillers to PVC mixtures such as Calcium Carbonate and Talc with different particles size at different amounts (5, 10 and 20 phr) were mechanically and thermally studied to find that, the suitable type of filler which can be used in enhancing the Vicat softening point, where it was Talc with particles size 0.7 Micron. By preparing the two best results achieved with PVC mixture which include PMMA and 0.7 Micron Talc it was obtained the highest Vicat softening point (VSP). It reached to 80.6 oC where it was 70.5 oC for the PVC without VSP improver and reached to 77.1 oC using commercial VSP improver.

Keywords: PVC Mixtures, Vicat Softening Point, Blended Polymers, composite, Inorganic Fillers, Calcium Carbonate.

1. Introduction

One of the challenges constraining the wide use of rigid PVC in many applications is its low heat resistance, whereas higher temperatures would cause softening and distortion for end products' dimensions. So, it is only recommended to be used in applications exposed to temperatures below 70°C.^[1]

Examples for some applications that PVC is limited to be used in:^[2,3] Rigid PVC pipes and fittings which used for transferring waste hot water like in Washing Machines for dishes and clothes.

Colored rigid PVC parts in building constructions like building panels, Window Profiles, Floor and Wall coverings which are directly exposed to sunlight especially in hot countries.

One of the suitable solutions for this problem is using additives like inorganic fillers where Calcium Carbonate and Talc can be used to improve Vicat softening point of PVC mixtures, but to very limited degrees.^[4-6] Or blending the PVC with commercial polymer heat modifiers like Alpha Methyl Styrene-Acrylonitrile (AMSAN) which can improve strongly Vicat softening point of PVC mixtures.^[7-12] Such kind of copolymer has been employed since 1970s, but there is limitation to be used due to its high price level comparing to PVC.^[13] AMSAN was chosen for improving Vicat softening point based on its high Tg comparing to PVC and the possible rates of increase are about 4°C per 10 % by weight of added AMSAN in PVC mixtures. Based on Tg value, Acrylonitrile

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Butadiene Styrene (ABS) which is well-known by its toughness and high ductility can be used for improving the Vicat softening point of PVC mixtures. ABS caused limited increasing in the heat stability of PVC mixtures, that were only in the range of 1.4 – 3.1 °C.^[14] Also, Acrylonitrile styrene acrylate (ASA) was presented in about 1970 as ABS replacement material with better weather resistance. ASA can be used as a cap layer for PVC to prevent color shift, especially yellowing, on exposure to sunlight and it has been shown that ASA can also be blended with PVC resulting in an increase in heat deflection temperature (HDT) and the relationship is linear to the amount of ASA polymer added.^[15,16] While Poly (methyl methacrylate) (PMMA) which is an ideal replacement of glass because of its high transparency and light weight can be blended with PVC to enhance the mechanical and thermal properties of PVC mixtures.^[19] The specific interaction between PMMA and PVC is almost as strong as hydrogen bonding. The DMA and SEM analysis confirmed the miscibility of this pair of polymers up to about 50wt% of PMMA. PVC/PMMA blend is compatible and the Tg of PVC/PMMA blends increased by linear relation as PMMA ratio increased.^[17-21]

2. Experimental

2.1. Materials

Suspension polyvinyl chloride (PVC) of K-value 56 was supplied from Shintech company; USA to be used as main polymer. Alpha Methyl Styrene Acrylonitrile copolymer was supplied from Galata Chemicals company; USA. Acrylonitrile Butadiene Styrene terpolymer (ABS) was supplied from Taita Chemical company, China, Acrylonitrile Styrene Acrylate terpolymer was supplied from LG Chemicals company; South Korea and Poly (Methyl methacrylate) polymer was supplied from LG Chemicals company; South Korea. These polymers as blended polymers with PVC. Two different particles size from calcium carbonate materials were used as inorganic filler in PVC mixtures, one was Hydrocarb 95T-OG with median diameter (d 50) 0.8 Micron from Omya company; France and the other was ZC 1T with median diameter (d 50) 2.6 Micron from ElZain Chemicals company, Egypt, and two different mean particles size Talc materials were used as another inorganic filler in PVC mixtures, one was Jetfine 0.7 C A with median diameter (d 50) 0.7 Micron from Imerys Talc company; France and the other type was

Luzenac A7 C with Mean particle size (d 50) 2.3 Micron from Imerys Talc company; France. Octyl-tin mercaptide stabilizer with grade name Mark 17 MOK A from Galata Chemicals GmbH, Germany was used as main stabilizer and epoxy soya bean oil (ESBO) with grade name CCP STAB CIZER B22R from Adeka Palmarole company; France was used as co-stabilizer. Medium molecular weight acrylic polymer with grade name KANE ACE PA-210 from Kaneka company; Japan was used as processing aid. Paraffin wax with grade name Sasolwax 6530 SA from Sasol Chemicals company; South Africa and oxidized polyethylene wax with grade name A-C 629A from Honeywell company; USA plus Calcium Stearate with grade name CA-30 from Tepekimya; Turkey were used as lubricant for prepared PVC mixtures. Titanium dioxide with grade name 2220 from Kronos; USA was used as a white pigment for coloring prepared PVC mixtures.

2.2. Preparations and Experimental Techniques

PVC mixtures were prepared by using industrial dry PVC batch mixer with capacity 600 L per batch and the mixing process was done by heating PVC mixture in the hot vessel to reach temperature 120 °C, then cooling to 40 °C in the cold vessel and for obtaining a good homogeneity PVC mixtures pelletized through PVC pelletizing machine containing twin screws at temperature 180 - 200 °C.

Testing samples prepared by injection molding process through injection molding machine with clamp force 220 ton where the injection temperature was around 180 °C.

Different polymer additives (AMSAN, ABS, ASA and PMMA) were blended with at different ratios with PVC polymer and the formulations of prepared PVC mixtures were as the following table 1:

From table 1, it was obvious that, the number of formulations was 13 formulations, the first was blank (No. 1), and the remained formulations were 3 formulations for each blended polymers at ratios 5, 10 and 20 % respectively. Formulations No. 2, 3 and 4 represented PVC/AMSAN blends. While, formulations No. 5, 6 and 7 represented PVC/ABS blends. As well as formulations No. 8, 9 and 10 represented PVC/ASA blends. And formulations No. 11, 12 and 13 represented PVC/PMMA blends.

Also, Different inorganic fillers (CaCO₃ and Talc) with different particle size (2.6-Micron CaCO₃, 0.8-Micron CaCO₃, 2.3-micron Talc and 0.7-Micron Talc)

were used as additives at different ratios to PVC resin in PVC mixtures and the formulations of prepared PVC mixtures were as following in table 2:

From table 2, it is obvious that, the number of formulations were 13 formulations, where the first was blank, and the remained formulations were 3 formulations for each particle size of added inorganic filler, where, formulations No. 14, 15 and 16 represented adding 2.6 Micron CaCO_3 and formulations and No. 17, 18 and 19 represented adding 0.8 Micron CaCO_3 , while, formulations No. 20, 21 and 22 represented adding 2.3 Micron Talc and formulations No. 23, 24 and 25 represented adding 0.7 Micron Talc.

2.3. Machines and Lab. Devices

2.3.1. Samples preparing machines

PVC Mixtures were prepared as a dry powder by using PVC batch mixer model COMBIMIX-HC/600/1500/FV, Plasmec Srl., Italy, then pelletized by using PVC pelletizing machine model KMD130-26G, KraussMaffei Technologies GmbH, Germany.

The prepared PVC mixtures pellets were injected by using injection molding machine model V-220, ENGEL GmbH, Austria to produce PVC fittings (with diameter 75 mm) for testing samples.

2.3.2. Lab testing devices

Tensile strength and elongation at break were measured with a Universal testing machine model Zwick DO-FB010TN, ZwickRoell, Germany at a crosshead speed of 5 mm/min according to ISO 6259-1/2015. Charpy impact strength was measured with a pendulum impact tester device model BPI-50COMC, IPT, Germany according to ISO 179-1/2010 at room temperature. The DSC and TGA of prepared PVC mixtures were analyzed in Clariant Company, Turkey; by using a differential scanning calorimeter model DSC 3, Mettler-Toledo AG, Switzerland with a heating rate of $10\text{ }^\circ\text{C min}^{-1}$. Vicat softening point (VSP) was measured with a Vicat tester device model IPT-1399, IPT, Germany according to ISO 2507/1995.

Table 1: PVC blended mixtures formulations

Row Materials	Blank No.1	PVC mixtures from No. 2 – No. 13		
		5% blending polymer	10% blending polymer	20% blending polymer
PVC	86.700	81.700	76.700	66.700
Blending Polymers AMSAN / ABS / ASA / PMMA	0	5	10	20
ESBO	1.100	1.100	1.100	1.100
TiO ₂	5	5	5	5
Processing aid	1.600	1.600	1.600	1.600
Tin Stabilizer	3.600	3.600	3.600	3.600
Paraffin Wax	0.900	0.900	0.900	0.900
Oxidized PE wax	0.100	0.100	0.100	0.100
Calcium Stearate	1	1	1	1
Total	100	100	100	100

Table 2: PVC mixtures formulations containing inorganic fillers

Row Materials (g)	PVC mixtures			
	Blank No.1	5 phr filler	10 phr filler	20 phr filler
PVC	86.700	82.350	78	69.350
Inorganic filler (CaCO_3 / Talc)	0	4.350	8.700	17.350
ESBO	1.100	1.100	1.100	1.100
TiO ₂	5	5	5	5
Processing aid	1.600	1.600	1.600	1.600
Tin Stabilizer	3.600	3.600	3.600	3.600
Paraffin Wax	0.900	0.900	0.900	0.900
Oxidized PE wax	0.100	0.100	0.100	0.100
Calcium Stearate	1	1	1	1
Total	100	100	100	100

3. Results and discussion:

First, the mechanical properties of the prepared blended PVC mixtures such as tensile strength, elongation at break and impact resistance were studied and the obtained results were as the following:

3.1. Effect of the presence of different blended polymers in PVC mixtures on Tensile Strength

Tensile strengths for prepared PVC mixtures from No. 1 to No. 13 were tested and the obtained results are illustrated in table 3.

3.2. Effect of the presence of different blended polymers in PVC mixtures on Elongation at break (%)

Elongation at break percentage results for the prepared PVC mixtures No. 1 to No. 13 were determined and the obtained results can be illustrated in table 4.

3.3. Effect of the presence of different blended polymers in PVC mixtures on Pendulum Impact resistance

Pendulum impact resistance for prepared PVC mixtures No. 1 to No. 13 are tested and the obtained results are illustrated in table 5.

Also, the thermal properties of the prepared blended PVC mixtures such as DSC, TGA and Vicat Softening point were studied and the obtained results were in table 5.

3.4. DSC for polymers blended PVC mixtures

From the results of DSC, the TG for blended PVC mixtures were measured and it were as in table 6

3.5. Effect of the presence of different blended polymers in PVC mixtures on Vicat softening point

Vicat Softening Point for prepared PVC mixtures from No. 1 to No. 13 and the results are illustrated in table 7:

Manufacturing properties for prepared PVC mixtures which blended with different polymers (AMSAN, ABS, ASA and PMMA) were examined and it was found that, they were processed easily within the processing window parameters of PVC and the surface finish was good which indicating the compatibility of these polymers with PVC.

Second, the mechanical properties of the prepared PVC mixtures containing inorganic filler (2.6 Micron CaCO₃, 0.8 Micron CaCO₃, 2.3 Micron Talc and 0.7

Micron Talc) such as tensile strength, elongation at break and impact resistance were studied and the results were as follows:

3.6. Effect of adding different amounts of inorganic fillers to PVC mixtures on Tensile Strength

Tensile strengths for prepared PVC mixtures No. 14 to No. 25 are illustrated in table 8:

3.7. Effect of adding different amounts of inorganic fillers to PVC mixtures on Elongation at break percentage

The results of elongation at break percentage for prepared PVC mixtures from No. 14 to No. 25 are illustrated in table 9:

3.8. Effect of adding different amounts of inorganic fillers to PVC mixtures on Pendulum Impact resistance

Pendulum Impact resistance for prepared PVC mixtures No. 14 to No. 25 are studied and the results are illustrated in table 10:

Also, the thermal properties of the prepared PVC mixtures containing inorganic filler (2.6 Micron CaCO₃, 0.8 Micron CaCO₃, 2.3 Micron Talc and 0.7 Micron Talc) such as DSC, and Vicat Softening point were studied and the obtained results are as follows:

3.9. DSC for filled PVC mixtures

Only two filled PVC mixtures (20 phr 0.8-micron CaCO₃ and 20 phr 0.7-micron Talc) were studied as a reference for the DSC. The following figures (21 and 22) represented DSC results for the studied PVC filled mixtures:

3.10. Effect of adding different amounts of inorganic fillers to PVC mixtures on the Vicat softening point

Effect of adding different amounts from the fillers (CaCO₃ and Talc) in VSP was studied and the results are shown in Table 11.

Manufacturing properties of prepared PVC mixtures which containing inorganic fillers with different particles size at different ratios were examined and it was found that they were processed easily within the processing window parameters of PVC and the surface finish was good which indicating the good dispersion of these fillers in these PVC mixtures.

Table 3: Tensile Strength for prepared PVC mixtures (No. 1 to No. 13)

Mixture No.	Blended Polymer With PVC	Tensile Strength Kg/cm ²	Mixture No.	Blended Polymer With PVC	Tensile Strength Kg/cm ²
1	Blank	558.07	8	5 % ASA	511.94
2	5 % AMSAN	595.00	9	10 % ASA	492.06
3	10 % AMSAN	590.00	10	20 % ASA	486.98
4	20 % AMSAN	584.00	11	5 % PMMA	527.32
5	5 % ABS	473.22	12	10 % PMMA	513.41
6	10 % ABS	495.18	13	20 % PMMA	618.68
7	20 % ABS	483.97			

Table 4: Elongation at break percentage for prepared PVC mixtures from No. 1 to No. 13

Mixture No.	Blended Polymer With PVC	Elongation at Break (%)	Mixture No.	Blended Polymer With PVC	Elongation at Break (%)
1	Blank	27	8	5 % ASA	29
2	5 % AMSAN	64	9	10 % ASA	24
3	10 % AMSAN	49	10	20 % ASA	32
4	20 % AMSAN	21	11	5 % PMMA	20
5	5 % ABS	25	12	10 % PMMA	18
6	10 % ABS	30	13	20 % PMMA	13
7	20 % ABS	25			

Table 5: Pendulum impact resistance values for prepared PVC mixtures (No. 1 to No. 13)

Mixture No.	Blended Polymer with PVC	Impact Value KJ/m ²	Mixture No.	Blended Polymer with PVC	Impact Value KJ/m ²
1	Blank	63.33	8	5 % ASA	222.88
2	5 % AMSAN	220.40	9	10 % ASA	154.60
3	10 % AMSAN	210.10	10	20 % ASA	157.01
4	20 % AMSAN	206.00	11	5 % PMMA	119.27
5	5 % ABS	140.32	12	10 % PMMA	82.93
6	10 % ABS	160.47	13	20 % PMMA	78.17
7	20 % ABS	282.53			

Table 6: DSC for polymers blended PVC mixtures

Type and amount of polymer	T _g °C	Type and amount of polymer	T _g °C
1-Blank PVC	82.02	ASA	
AMSAN	-	8- 5%	82.40
2- 5%	82.02	9- 10%	82.66
3- 10%	85.48	10- 20%	84.80
4- 20%	85.94	PMMA	
ABS	-	11- 5%	81.94
5- 5%	-	12- 10%	8.97
6- 10%	84.00	13- 20%	-
7- 20%	84.00		

Table 7: Vicat Softening point for prepared PVC mixtures from No. 1 to No. 13

Mixture No.	Blended Polymer With PVC	Vicat Softening Point (°C)	Mixture No.	Blended Polymer With PVC	Vicat Softening Point (°C)
1	Blank	72.50	8	5 % ASA	72.95
2	5 % AMSAN	75.00	9	10 % ASA	74.20
3	10 % AMSAN	76.30	10	20 % ASA	76.35
4	20 % AMSAN	77.10	11	5 % PMMA	73.50
5	5 % ABS	73.90	12	10 % PMMA	75.35
6	10 % ABS	74.30	13	20 % PMMA	78.60
7	20 % ABS	75.10			

Table 8: Tensile Strength for prepared PVC mixtures (No. 14 to No. 25)

Mixture No.	Filler Amounts	Tensile Strength Kg/cm ²	Mixture No.	Filler Amounts	Tensile Strength Kg/cm ²
1	Blank (Zero)	558.07	20	5 phr 2.3 M Talc	516.15
14	5 phr 2.6 M CaCO ₃	534.88	21	10 phr 2.3 M Talc	552.37
15	10 phr 2.6 M CaCO ₃	502.91	22	20 phr 2.3 M Talc	571.67
16	20 phr 2.6 M CaCO ₃	481.36	23	5 phr 0.7 M Talc	552.80
17	5 phr 0.8 M CaCO ₃	426.06	24	10 phr 0.7 M Talc	554.56
18	10 phr 0.8 M CaCO ₃	477.82	25	20 phr 0.7 M Talc	572.18
19	20 phr 0.8 M CaCO ₃	464.59			

Table 9: Elongation at break percentage for prepared PVC mixtures No. 14 to No. 25

Mixture No.	Filler Amounts	Elongation at Break (%)	Mixture No.	Filler Amounts	Elongation at Break (%)
1	Blank (Zero)	27	20	5 phr 2.3 M Talc	27
14	5 phr 2.6 M CaCO ₃	20	21	10 phr 2.3 M Talc	18
15	10 phr 2.6 M CaCO ₃	21	22	20 phr 2.3 M Talc	8
16	20 phr 2.6 M CaCO ₃	17	23	5 phr 0.7 M Talc	24
17	5 phr 0.8 M CaCO ₃	10	24	10 phr 0.7 M Talc	16
18	10 phr 0.8 M CaCO ₃	25	25	20 phr 0.7 M Talc	11
19	20 phr 0.8 M CaCO ₃	27			

Table 10: Pendulum impact resistance values for prepared PVC mixtures (No. 14 to No. 25)

Mixture No.	Filler Amounts	Impact Value KJ/m ²	Mixture No.	Filler Amounts	Impact Value KJ/m ²
1	Blank (Zero)	63.33	20	5 phr 2.3 M Talc	89.00
14	5 phr 2.6 M CaCO ₃	82.22	21	10 phr 2.3 M Talc	35.84
15	10 phr 2.6 M CaCO ₃	94.77	22	20 phr 2.3 M Talc	30.23
16	20 phr 2.6 M CaCO ₃	96.01	23	5 phr 0.7 M Talc	74.66
17	5 phr 0.8 M CaCO ₃	82.17	24	10 phr 0.7 M Talc	63.96
18	10 phr 0.8 M CaCO ₃	91.29	25	20 phr 0.7 M Talc	51.96
19	20 phr 0.8 M CaCO ₃	97.96			

Table 11: Vicat Softening point for prepared PVC mixtures No. 14 to No. 25

Mixture No.	Filler Amounts	Vicat Softening Point (°C)	Mixture No.	Filler Amounts	Vicat Softening Point (°C)
1	Blank (Zero)	72.5	20	5 phr 2.3 M Talc	72.60
14	5 phr 2.6 M CaCO ₃	72.5	21	10 phr 2.3 M Talc	74.45
15	10 phr 2.6 M CaCO ₃	72.6	22	20 phr 2.3 M Talc	75.30
16	20 phr 2.6 M CaCO ₃	72.8	23	5 phr 0.7 M Talc	74.45
17	5 phr 0.8 M CaCO ₃	72.7	24	10 phr 0.7 M Talc	74.90
18	10 phr 0.8 M CaCO ₃	72.8	25	20 phr 0.7 M Talc	75.65
19	20 phr 0.8 M CaCO ₃	73.0			

3.11. Optimum PVC mixture which had the highest Vicat softening point

From the previous data, it could be observed that the highest Vicat softening point found in PVC mixture No. 13 which contained 20% PMMA and PVC mixture No. 25 which contained 20 phr 0.7-micron talc.

Finally, PVC mixture No.26 was prepared by blending 20% PMMA and filled with 20 phr 0.7 Micron Talc with PVC and the following table 12 showed PVC mixture No.26 composition in details:

It was found that PVC mixture No. 26 had the

highest Vicat Softening point reach to 80.6 °C and it proved by examining DSC where it was 89.15°C.

The following figure 1 represented DSC results for PVC mixture No.26: (where the Tg of this formulation was determined).

The mechanical and thermal properties of formulation No: 26 was represented in table 13

It was processed easily within the processing window parameters of PVC and the surface finish was good which proved the compatibility of PMMA with PVC and good dispersion of 0.7 Micron Talc in PVC mixture.

Table 12: PVC mixture No.26

Row Materials	PVC mixture No.26
PVC	52.800
PMMA	20.000
0.7-Micron Talc	13.900
ESBO	1.100
TiO ₂	5.000
Processing aid	1.600
Tin Stabilizer	3.600
Paraffin Wax	0.900
Oxidized PE wax	0.100
Calcium Stearate	1.000
Total	100

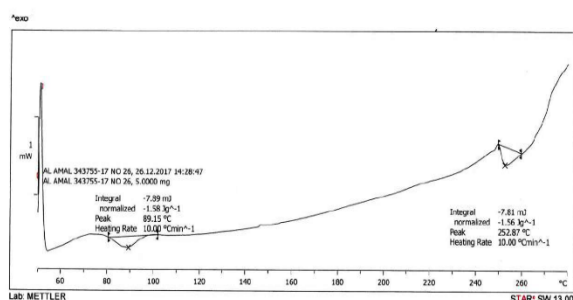


Fig. 1 DSC study results for the formulation No 26

Table 13: Mechanical and thermal properties of the final PVC mixture. (No.26)

Mechanical Properties	Unit	Value
Tensile Strength	Kg/cm ²	607.36
Tensile Elongation at break	%	11.85
Pendulum Impact	KJ/m ²	25.15
T _g	°C	89.15
Vicat softening temperature	°C	80.60

4. Conclusions

Comparing the mechanical, thermal and manufacturing properties of successfully prepared PVC mixtures based on blending technique using different types of polymers (ABS, ASA and PMMA) with PVC mixtures containing commercial polymer heat modifier AMSAN. It was found that the addition of ABS or ASA to PVC mixtures strongly reduced the tensile strength of the mixture. It was the same for PMMA. Also, it was observed that, the optimum result of elongation was achieved by blending PVC with 5% of AMSAN in PVC mixture. The most important notice that, the addition of AMSAN, ABS, ASA or PMMA to PVC mixtures enhanced the Vicat softening point and the optimum result of Vicat softening point was achieved by blending PVC with 20% of PMMA in PVC mixture. Optimum result of tensile strength was achieved by adding 20 phr of 0.7 Micron Talc to PVC mixture, while the addition of 2.3 Micron Talc to

PVC mixtures increased the pendulum Impact resistance. The optimum result of pendulum impact resistance was achieved by adding 20 phr of 0.8 Micron CaCO₃ to PVC mixture. As well as, it was noticed that, the addition of 2.6 Micron or 0.8 Micron CaCO₃ and 2.3 Micron or 0.7 Micron Talc to PVC mixtures enhanced the Vicat softening point and the optimum result of Vicat softening point was achieved by adding 20 phr of 0.7 Micron Talc to PVC mixture. Finally, it was concluded that, by using blending technique to add 20% PMMA as polymer heat modifier and 20 phr 0.7 Micron Talc as inorganic filler to PVC polymer enhanced the Vicat softening point of this PVC mixture and to reach 80.6 °C.

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