



PREPARATION AND FTIR STUDIES OF PMMA/PVC POLYMER BLENDS, PVC-g-PMMA GRAFT COPOLYMERS AND EVALUATING GRAFT CONTENT[#]

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Abstract. This study presents the qualitative and quantitative analyses of Fourier Transform Infrared (FTIR) spectra of poly(methyl methacrylate)/poly(vinyl chloride) blends (PMMA/PVC), as well as PVC-g-PMMA graft copolymers. Graft copolymerizations of methyl methacrylate (MMA) onto PVC macromolecules were carried out in the mixture of cyclohexanone/N,N-dimethylformamide as solvents, dibenzoyl peroxide as initiator and nitrogen medium. FTIR spectra of PMMA/PVC blends showed that there were molecular interactions between C=O groups of PMMA and C-Cl groups of PVC. Assignments of infrared absorption bands for specific groups of PMMA/PVC blends have been contributed. Using regression method, linear calibration curve between PMMA mole content and peak areas of C=O groups in FTIR spectra of the blends has been found when C=O peak areas were multiplied by an appropriate factor. PMMA grafted content and total PMMA formed content in PVC-g-PMMA graft copolymers have been evaluated. The results showed that grafted PMMA content was increased since PVC was initially dechlorinated by NaOH solution. The grafted PMMA content and total formed PMMA content were 5.05 wt.% and 11.25 wt.%, respectively, when MMA monomers were grafted onto neat PVC and modified PVC molecules.

Keywords: graft copolymer, PVC-g-PMMA, grafted content, PMMA/PVC blends.

Classification numbers: 2.9.3, 2.10.2.

1. INTRODUCTION

Poly(methyl methacrylate) (PMMA) is widely used in various fields such as optical sensors, optical windows, biomaterials, molecular separations, etc. [1, 2]. It has been found that plasticized PMMA could be used as gel electrolytes for lithium batteries [3, 4]. The possibility

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of using plasticized PMMA-based polymer blends as gel electrolytes have been also explored [5-9]. Among them, poly(vinyl chloride) (PVC) is more often used for improving the poor mechanical properties of PMMA gel when using with high level of plasticizers. The plasticized PMMA/PVC blends have been received much attention because they are promised materials for polymeric electrolyte application. Some studies have showed that the miscibility of PMMA/PVC blends depends on molecular weights and proportion of each component [8-14]. Therefore, using of a compatibilizer can further improve properties of the PMMA/PVC blends. Recently, Li et al. [11] have synthesized poly(methyl methacrylate – *graft* – polyvinylchloride) (PMMA-*g*-PVC) for blending with PMMA in order to prepare dual-phase polymer electrolytes, in which PMMA grafted content was evaluated by proton nuclear resonance spectroscopy with the value of 6.1 weight percent (wt.%).

In this study, we provide another synthetic route for preparing PVC-*g*-PMMA graft copolymer with higher grafted content. Fourier transform infrared (FTIR) spectroscopy was also used as a method for evaluating graft content. From FTIR quantitative analysis of PMMA/PVC blends, a calibration curve which describes the relationship between infrared absorbance of carbonyl groups of PMMA against PMMA mole content in the blends was set up. Therefore, the PMMA grafted content can be known easily. The graft copolymer can be prospectively used as a compatibilizer for plasticized PMMA/PVC blend electrolytes. Moreover, due to some differences in band assignments in previous studies of PMMA/PVC blends [12-16], this study was also attempted to contribute to the band assignments in middle infrared range for the blends and their molecular interactions.

2. EXPERIMENTAL

2.1. Materials

PVC (SG-660 type, k index of 65-67) was supplied by TPC Vina Plastic & Chemical Corp., Dong Nai, Vietnam. PMMA ($M_w = 120000$) was purchased from Aldrich (USA). Methyl methacrylate (MMA, 99 %, Sigma Aldrich, contained 30 ppm MEQH) was passed through basic alumina to remove MEQH inhibitor right before every experiment [17]. Tetrahydrofuran (THF), cyclohexanone, N,N dimethyl formamide (DMF) and acetone were analytical grade products (AG) of Xilong Scientific Co., Ltd. (China). Absolute ethanol and methanol alcohols were purchased from Duc Giang Chemicals and Detergent Powder Joint Stock Company (Vietnam). Above solvents were used as received without further purification. Dibenzoyl peroxide (BPO, Xilong) was purified by dissolving in acetone, filtering, precipitating with addition of absolute methanol; white crystalline powder was obtained and dried in a vacuum oven for 48 h at ambient room temperature.

2.2. Synthetic procedures

2.2.1. Synthesis of PVC-*g*-PMMA

PVC (3.0 g), cyclohexanone (24 mL) and DMF (3 mL) were charged in a 100 mL two-neck flask over a magnetic stirrer. After homogeneous solution was obtained, three drops of 1 wt.% thymolphthalein in absolute ethanol as an indicator were added and the solution was continuously purged with nitrogen gas. PVC was partially dechlorinated (0.31 mol.%) by adding 0.5N NaOH solution (0.3 mL) via a syringe, the light yellow-colored solution was gradually observed due to the formation of conjugated double bonds. The pH indicator showed that NaOH

was consumed completely corresponding to the disappearance of pink color. MMA monomers (1.0 g) and BPO initiator (0.08 g) were added via a syringe through side neck, nitrogen purging was continued for 15 minutes before sealing the flask. The flask was placed in an oil bath with controlled temperature of 60 ± 2 °C. Reaction was performed for 6 hours and terminated by immersing the flask in cold water. The product of PVC-g-PMMA (named PVCg1) was obtained by precipitation into excess 20-fold volume of methanol, following washed 3 times with methanol and dried in a vacuum oven for 24 h at 40 °C. The similar grafting procedure was also performed with neat PVC (PVCg0). PMMA homopolymer was extracted out of PVCg0 and PVCg1 samples by soxhlet with acetone for 48 hours. The final obtained solid products were named as PVCg0ex and PVCg1ex, respectively.

2.2.2. Preparation of PMMA/PVC blend films

Films of PMMA/PVC blends were prepared by casting method. Two solutions of PMMA and PVC were prepared by dissolving them in THF solvent in two separated Erlenmeyer flasks with the same concentration of 5 wt.%. Weights of PMMA and PVC solutions, as indicated in Table 1, were mixed in a vial, stirred for 1 hour and cast on glass plates. Evaporation of THF solvent was allowed at room temperature in a fume hood for 24 hours, followed by drying in vacuum oven at 40 °C for 24 hours. Seven PMMA/PVC blend samples with different weight ratios were obtained and stored in a desiccator.

Table 1. Weights of PMMA and PVC (W_{PMMA} , W_{PVC}) solutions for preparing PMMA/PVC blends.

Sample code	W_{PMMA} (g)	W_{PVC} (g)	PMMA/PVC (wt./wt.)	PMMA/PVC (mol./mol.)
B1	4.0	-	100/0	100/0
B2	3.2	0.8	80/20	75/25
B3	2.6	1.4	65/35	54/46
B4	2.0	2.0	50/50	38/62
B5	1.4	2.6	35/65	25/75
B6	0.8	3.2	20/80	14/86
B7	-	4.0	0/100	0/100

2.3. FTIR study and titration method

FTIR spectra of PMMA/PVC blends and PVC-g-PMMA samples were carried out using a Nexus Nicolet 670 spectrometer (USA) at room temperature, in the range of 4000-500 cm^{-1} with resolution of 4 cm^{-1} and averaging of 32 scans. In this studies, vibration modes are commonly labelled with lower-case Greek letters, such as: stretching, asymmetric, symmetric, out-of-plane stretching modes (ν , ν_{asym} , ν_{sym} , ν_{oop}); bending, in phase bending, out of phase bending (δ , δ_{ip} , δ_{op}), rocking, wagging and twisting (γ_{r} , γ_{w} , γ_{t}). For quantitative analysis, areas of peaks under FTIR spectra were evaluated by using Omnic software version 5.2a with the same spectral manipulations.

Calibration curve was made based on relationships between peak areas of $\delta(\text{C}=\text{O})$ in FTIR spectra and the known PMMA mole content of PMMA/PVC blends. PMMA content (including PMMA homopolymer) and grafted PMMA content in PVC-g-PMMA was then evaluated from this curve.

3. RESULTS AND DISCUSSIONS

3.1. Groups assignments and molecular interactions in PMMA/PVC blends

Figure 1 displays FTIR spectra of neat PMMA, neat PVC and PMMA/PVC blends. Band assignments for specific groups in PMMA, PVC, PMMA/PVC blends have been already reported in the literature [12, 14, 18-23]. Regarding to some different assignments, Table 2 presents band assignments for PMMA/PVC blends which are based on previous theoretical and experimental studies on neat PMMA, PVC [24-30], in combination with our experimental FTIR study.

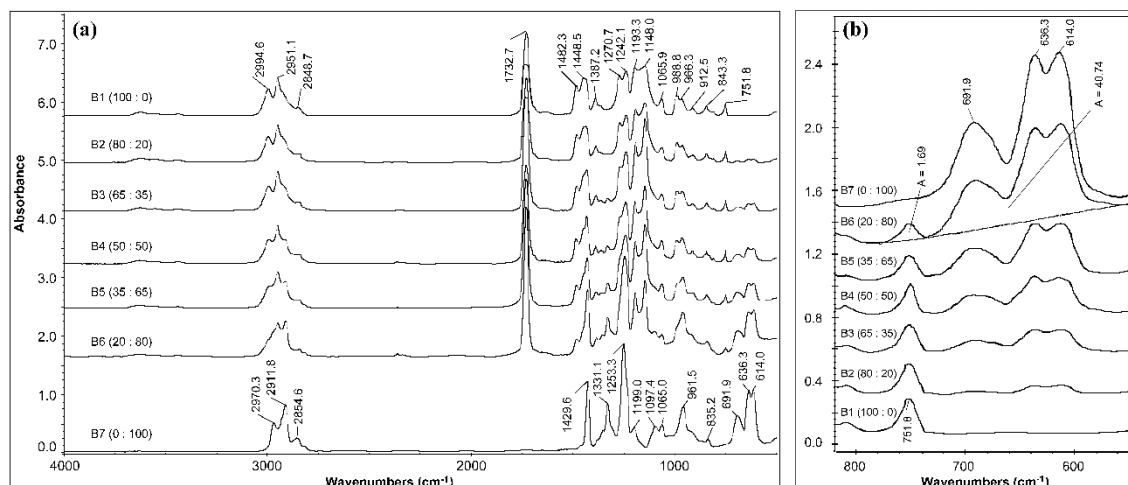


Figure 1. FTIR spectra of neat PMMA, PVC and their blends with different fraction weight ratios.

In spectra of PMMA/PVC blends, some peaks are unchanged in comparison with neat PMMA and neat PVC, however, some bands are overlapped or absent and shifted to another positions. The first overlap band occurs in the region 2800-3100 cm⁻¹ between the vibration band of $\nu(\text{CH}_3)_{\text{asym}}$ (at 2995 cm⁻¹ for neat PMMA) and the vibration band of $\nu(\text{CH})_{\text{op}}$ (at 2970 cm⁻¹ for PVC). The resulting bands are either a small shoulder at 2970 cm⁻¹ for blend B6 or new peaks appeared at wavenumbers between 2970 and 2995 cm⁻¹ for the blends B5-B3 (Table 2 Entry 1). The second overlap band occurs between vibration mode of $\delta(\text{OCH}_3)_{\text{ip}}$ (at 1449 cm⁻¹ for neat PMMA) and vibration mode of $\delta(\text{CH}_2)$ (at 1430 cm⁻¹ for neat PVC), leading to form a new peaks at position between 1430 cm⁻¹ and 1449 cm⁻¹ (Table 2 Entry 5). Similar overlap also happened with the peak at 1253 cm⁻¹ ($\delta(\text{CH})_{\text{ip}}$ in neat PVC) and the doublet peaks at (1271; 1242) cm⁻¹ ($\nu_{\text{asym}}(\text{C}-\text{C}-\text{O})$ in neat PMMA). The resulting bands (Table 2 Entry 7) are either single peaks (for the blends B4-B6) or new doublets centered between 1271-1253 cm⁻¹ (for the blends B2-B3). In the region 1025-925 cm⁻¹, there is an overlap band between 2 peaks of OCH₃ groups with peak of HCCl groups. The resulting band has a single peak (at 963 cm⁻¹ for the blend B6) or two peaks (for the blends B5-B2), meaning that one of three peaks is quite disappeared. The peaks of $\delta(\text{OCH}_3)_{\text{ip}}$ shift to lower wavenumbers, meanwhile the other shift to higher wavenumbers (Table 2 Entry 10).

It is noted that there are hydrogen bonds between HC-Cl groups in the PVC phase (Cl-C-H...Cl-C-H). However, the absence of some characteristic bands of HC-Cl groups and the overlapping phenomena possibly arise from the formations of new hydrogen bonds (Cl-C-

H...O=C-O) and polar interactions (HC-Cl δ^- ... δ^+ C=O) between CHCl groups of PVC with O=C=O groups of PMMA in their blends [30].

It can be found that the peaks in region between 820-550 cm $^{-1}$ in FTIR spectra of PMMA/PVC blends are very important for quantitative analysis. This region was expanded and displayed in Figure 1(b). It can be seen that peak intensities of $\delta(\text{C=O})_{\text{op}}$ (at 751 cm $^{-1}$) and $\nu(\text{C-Cl})$ (at ~692, ~636, ~612 cm $^{-1}$) are increased with increasing PMMA and PVC content, respectively. When PMMA content is increased, the peak intensity of $\delta(\text{C=O})_{\text{op}}$ is increased, in accordance with the peak intensity of $\nu(\text{C-Cl})$ and PVC content is decreased.

Table 2. FTIR assignments for specific groups in neat PMMA, neat PVC and PMMA/PVC blends.

Wavenumbers (cm $^{-1}$) of specific groups in PMMA/PVC blends (wt./wt.)									
Entry	Group	Vibration modes (in polymer)	100/0	80/20	65/35	50/50	35/65	20/80	0/100
1	CH ₃	ν_{asym} (PMMA) [12]	2995	2995	2994	2992	2989	-	-
	CHCl	ν_{oop} (PVC) [31]	-	-	-	-	-	2970 ^{sh}	2970
	CH ₂	ν_{asym} (PMMA) [24]	2951	2951	2951	2951	2951	2951	-
	CH ₂	ν_{asym} (PVC) [12, 28]	-	-	-	2914	2913	2912	2912
	CH ₂	ν_{sym} [22, 23]	2850	2850	2850	2845	2846	2846	2855
2	C=O	ν [24]	1733	1730.2	1730	1731	1730	1731	-
3	CH ₃	δ_{asym} [24]	1482	1482	1482	1485	1483	1482	-
4	CH ₂	δ [29, 32]	1449	1439	1436	1435	1433	1432	1430
5	αCH_3	δ_{sym} [24, 33, 34]	1387	1387	1387	1386	1386	1384	-
6	CH	δ_{op} [29, 33] (in PVC)	-	1331	1331	1331	1331	1331	1331
7	C-C-O	ν_{asym}	1271	1270	1268	-	-	-	-
	C-C-O	ν_{asym} [24]	1242	1242	1243	1243	1246	1249	-
	CH	δ_{ip} [33] (in PVC)	-	-	-	-	-	-	1253
8	CH ₃	γ_{w} [27]	1193	1193	1193	1193	1194	1194	-
	CH ₃	γ_{r} [27]	1148	1149	1149	1149	1149	1149	-
9	C-C	ν in amorphous) [24]	1066	1066	1066	1065	1066	1066	1065
		ν (in crystalline) [33]	-	-	-	1103	1101	1101	1097
10	OCH ₃	δ_{ip} [24]	989	989	988	987	985	-(sh)	-
	OCH ₃	γ_{r} [24]	966	966	965	966	964	963	-
	CHCl)	<i>Trans</i> - γ_{w} (PVC) [12]	-	-	-	-	-	-	962
11	CH ₃	γ_{r} [24, 27]	913	913	914	913	913	-	-
12	CH ₂	γ_{r} (PMMA) [24, 27]	843	843	843	842	842	842	-
	CH ₂	γ_{r} (PVC) [29]	-	-	-	-	-	-	835
13	C=O	δ_{oop} [24, 27]	752	752	752	750	752	751	-
14	C-Cl	ν [20, 35]	-	693	691	693	692	692	692
15	C-Cl	ν [35]	-	637	636	637	636	636	636
			-	612	612	612	612	613	614

3.2. FTIR quantitative analysis

Table 3 represents peak areas of C=O groups, C-Cl groups (A_{CO} , A_{CCl}) in the region of 820 - 550 cm $^{-1}$, instead of peak heights (for more accurate analysis) [36]. Consequently, percentages of A_{CO} over total peak areas of A_{CO} and A_{CCl} have been also calculated by the given formulas in Table 3. Table 3 shows that peak areas of C=O are increased with increasing of PMMA (or decreasing of PVC) mole content in the blends. From these results, Figure 2.a shows the variation of $y_1 = 100 \times A_{\text{CO}} / (A_{\text{CO}} + A_{\text{CCl}})$ as function of PMMA mole content (x). It can be seen

that this curve is non-linear (Figure 2.a). Therefore, A_{CO} was multiplied by a factor k before calculating $y_k = 100 \times k \times A_{CO} / (k \times A_{CO} + A_{CCl})$, (k was allowed to vary). Applying linear regression model, it has been found that R squared reached to maximum value of 0.9968, when $k = 3.4552$, and linear trend line of $y_k = 1.0111x$ can be obtained, as shown in Figure 2.b. The final regression line can be used as calibration curve (Figure 2.b).

Table 3. Peak areas of C=O groups and C-Cl groups from FTIR spectra of PMMA/PVC blends and other calculations.

Sample code	PMMA (x: mol.%)	A_{CO}	A_{CCl}	$y_1 = \frac{100 \times A_{CO}}{A_{CO} + A_{CCl}}$ (%)	$y_k = \frac{100 \times k \times A_{CO}}{k \times A_{CO} + A_{CCl}}$ (%)
B1	100.0	6.14	0.00	100.0	100.0
B2	74.66	3.43	3.16	52.05	79.08
B3	53.72	3.03	9.56	24.07	52.71
B4	38.46	2.73	15.96	14.63	36.32
B5	25.18	2.53	23.38	9.76	26.95
B6	13.51	1.69	40.47	3.98	11.42
B7	0.00	0.00	61.06	0.00	0.00

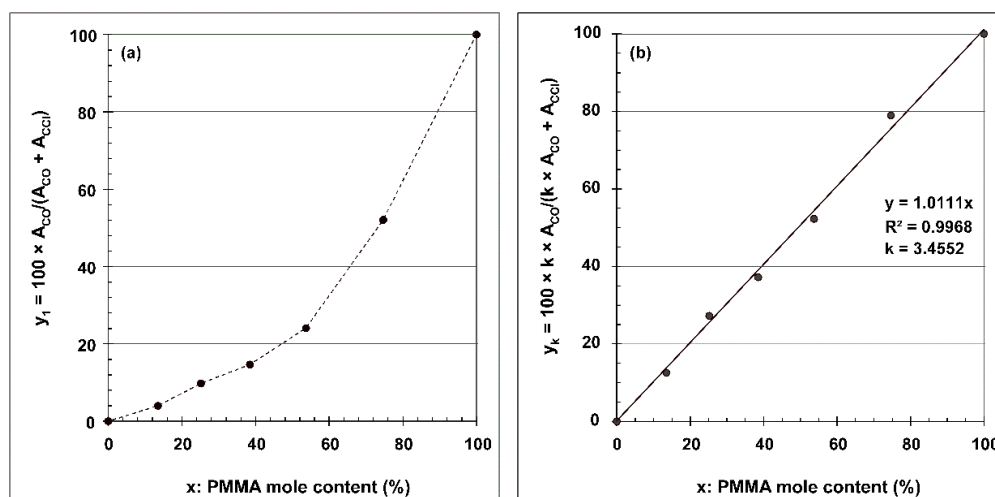


Figure 2. Percentages of $\delta(C=O)$ peak areas calculated from FTIR spectra of PMMA/PVC blends as functions of PMMA mole content.

3.3. FTIR spectra of PVC-g-PMMA copolymers

Figure 3.a displays FTIR spectra of neat PVC, modified PVC and extracted PVC-g-PMMA graft copolymers (PVCg0ex, PVCg1ex), Figure 3.b depicts the reaction scheme of grafting MMA monomers onto PVC chains to form graft copolymers. In spectra of modified PVC, two new peaks appear. The first is at 1725 cm^{-1} which is attributed to ketone groups. These groups can be formed at allylic chlorine sites due to the presence of oxygen during precipitation [37]. The band from $1680 - 1600 \text{ cm}^{-1}$ with the peaks centered at 1640 and 1607 cm^{-1} are attributed to C=C symmetric and asymmetric stretch vibrations in conjugated double bonds (as depicted in Figure 3.b-Eq.1). In spectra of PVCg0ex and PVCg1ex, there are some peaks at 1731 , 1194 , 1150 and 750 cm^{-1} which are attributed to $\nu(C=O)$, $\gamma_w(CH_3)$, $\gamma_t(CH_3)$ and $\delta(C=O)$. It is noted that PMMA homopolymer has been extracted out of the polymeric products of PVCg0 and PVCg1. This indicates PMMA chains have been successfully grafted onto PVC chains. In spectra of

PVCg1ex, there is the absence of the vinyl absorption bands in the region of 1640-1600 cm^{-1} . This testifies that the MMA monomers were reacted with PVC at these sites, as depicted in Eq.3 of Figure 3.b. At allylic chlorine, in the absence of oxygen, MMA can be also easily grafted.

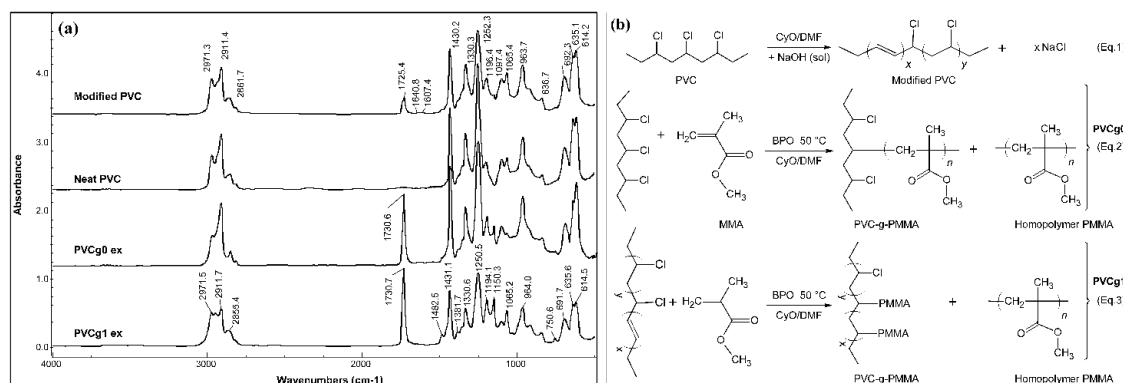


Figure 3. (a): FTIR spectra of neat PVC, modified PVC and extracted graft copolymers (PVCg0ex and PVCg1ex); (b): Reaction scheme of grafting MMA monomers onto PVC chains. (CyO: cyclohexanone).

3.4. Evaluation of grafted content in copolymers

Table 4 represents the results of total PMMA formed content (including PMMA homopolymer) and grafted PMMA content in the PVCg0 and PVCg1 graft copolymers. In the PVCg0, MMA monomers can graft onto PVC backbones to form PMMA side chains at random chlorine sites through BPO initiator (Eq.2), the mechanism can be seen in the reference [11]. In this case, the grafted PMMA content is only 3.23 mol.% or 5.05 wt.% and the total PMMA formed content is 7.81 mol.% or 11.93 wt.%.

In order to increase grafting efficiency, PVC was partially dechlorinated (or modified) by NaOH to form conjugated double bonds and allylic chlorine atoms which were expected as active centers for grafting of monomers (Eq.1). At these active sites, MMA can be grafted easily onto modified PVC chains, as illustrated in (Eq.3 of Figure 4). The obtained results show that PMMA grafted content PVCg1 is 7.34 mol.% or 11.25 wt.%. It means the PMMA grafted content in PVCg1 is about 2.3-fold increase in comparison with that of the PVCg0.

Table 4. Area peak calculations from FTIR spectra of PVC-g-PMMA graft copolymers.

Sample code	A_{CO}	A_{CCl}	y_k (%)	$x = \text{PMMA}$ (mol.%)	PMMA (wt.%)
PVCg0	0.973	39.24	7.89	7.81	11.93
PVCg0ex	0.276	28.30	3.26	3.23	5.05
PVCg1	1.605	34.33	13.91	13.75	20.33
PVCg1ex	0.981	42.12	7.42	7.34	11.25

4. CONCLUSIONS

FTIR spectra of PMMA/PVC polymer blends as well as PVC-g-PMMA graft copolymers have been investigated. Absorption bands of PMMA/PVC blends have been assigned. FTIR analytic results indicated that there were molecular interactions between C=O groups of PMMA and C-Cl groups of PVC. The relationship between PMMA mole content in the blends and its

peak areas of C=O deformation absorption can be used for making calibration curves. From these curves, PMMA grafted content as well as PMMA formed content can be evaluated in the PVC-g-PMMA graft copolymers. The modification of PVC via partial dechlorination can generate active sites which facilitate the grafting and improving graft efficiency of MMA monomers onto PVC chains.

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