# Quantitative Analysis of Copolymers and Blends of Polyvinyl Acetate (PVAc) Using Fourier Transform Infrared Spectroscopy (FTIR) and Elemental Analysis (EA)

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**Abstract** Fourier transform infrared spectroscopy (FTIR) is one of the most important instrumental techniques used to study the molecular structure of organic polymers. As part of our efforts to increase instrumental use in the undergraduate chemistry laboratory we have developed a quantitative FTIR experiment for use in our quantitative and instrumental analysis courses. The objective of the experiment is to determine the percent composition of PVAc in copolymers and blends with polyethylene (PE) and n-vinyl pyrrolidone (PVP). We report on the experimental methods used and the results obtained on examining the quantitative FTIR and elemental analysis of a series of ethylene-vinyl acetate (EVAc) copolymers. The EVAc copolymers ranged from 10% to 70% PVAc. Copolymers and blends of n-vinyl pyrrolidone-polyvinyl acetate (PVP/PVAc) ranging from 20% to 80% PVAc were also analyzed.

*Keywords*: *quantitative analysis, elemental analysis, Fourier Transform Infrared Spectroscopy, undergraduate laboratory experiment, hand-on learning, Ethylene-vinyl acetate copolymer, n-vinyl pyrrolidone - vinyl acetate copolymer and blends*

**Cite This Article:** Ronald P. D'Amelia, Samantha Gentile, William F. Nirode, and Ling Huang, "Quantitative Analysis of Copolymers and Blends of Polyvinyl Acetate (PVAc) Using Fourier Transform Infrared Spectroscopy (FTIR) and Elemental Analysis (EA)." *World Journal of Chemical Education*, vol. 4, no. 2 (2016): 25-31. doi: 10.12691/wjce-4-2-1.

## 1. Introduction

Infrared spectroscopy (IR) is a common instrumental technique that is taught in organic, analytical, and instrumental courses at many undergraduate institutions. It is routinely used for many qualitative and quantitative chemical analyses [1-9]. Today, Fourier transform infrared spectroscopy (FTIR) is one of the most important instrumental methods used to elucidate the compositions and molecular structures of organic polymeric compounds. There have been several published experiments and reports on determining the composition of copolymers in general [10,11,12,13,14] and specifically for copolymers of ethylene-vinyl acetate (EVAc) [15,16,17,18] using IR spectroscopy. Mathias and co-workers [16] utilizes FTIR transmission mode to measure for thin films of EVAc copolymers with quantification based on the ratio of absorbances at 1020 cm$^{-1}$ and 720 cm$^{-1}$ respectively. K. Williams [17] expands Mathias's experiment by attaching an ATR accessory to a FTIR spectrometer and combining the IR results with thermogravimetric data. There is also an ASTM method (D5594-98-revised 2004) [19] which covers IR procedures for determining the vinyl acetate content of EVAc copolymers using pressed film or molded plaques. This test method is applicable for the analysis of EVAc copolymers containing 0.5 to 55 % vinyl acetate. The vinyl acetate is measured using IR absorbance bands at 1020 cm$^{-1}$ (0.5 to 5% VA) or 609cm$^{-1}$ (5-55% VA). As part of our efforts to increase exposure to FTIR spectroscopy in the undergraduate chemistry laboratory curriculum we have developed a quantitative FTIR experiment for use in our analytical and instrumental courses. This experiment was developed as a teaching exercise illustrating the general principles of both qualitative and quantitative FTIR methodologies.

The basic principle of the FTIR technique is based on the fact that within any molecule/polymer the atoms or groups of atoms vibrate with a few definite, sharply defined frequencies characteristic of the compound. The IR frequencies are indicative of the transitions between vibrational energy states for specific bonds and types of vibrations and lie in the infrared region of the electromagnetic spectrum. When the sample is placed in the IR beam it absorbs energy at specific frequencies characteristic of the molecule and transmits all other frequencies. The resulting pattern is known as a molecular infrared vibrational absorption spectrum. The intensity of the absorption is a known reproducible function of the amount present.

The objective of our experiment is to determine the % composition of polyvinyl acetate (PVAc) in copolymers and blends with polyethylene (PE) and poly n-vinyl pyrrolidone (PVP). These copolymers were selected because they are all commercial copolymers of PVAC and are used in many everyday products. For example, ethylene-vinyl acetate (EVAC) copolymer has many varied uses in the home, sports equipment and for industrial and medical applications such as transdermal patches. They can be used as hot melt adhesives, hot glue sticks, soccer cleats and also can be used in some biomedical engineering applications as a drug delivery device. EVAC can range from a soft flexible material having high vinyl acetate (VA) content to a hard rigid substance having low VA. Copolymers of n-vinyl pyrrolidone and vinyl acetate monomers are of significant utility in the pharmaceutical and cosmetic industries. Applications include hair styling, skin care, membranes for medical devices, and some other uses for water treatment and as adhesives. The ratio of PE and PVP to PVAc can significantly affect the physical properties of the final product. Therefore, it is important to identify the specific composition of these copolymers.

## 1.1. Learning Objectives:

1. To record and analyze ATR-FTIR data on commercial polyvinyl acetate co-polymers of different compositions.

2. To prepare correlation curves of the absorbance ratio of specific peaks vs % composition of PVAc as determined by elemental analysis.

3. To determine the polyvinyl acetate content in an unknown sample of ethylene-vinyl acetate copolymers or copolymers of n-vinyl pyrrolidone and vinyl acetate.

## 2. Experimental Methods and Materials

This experiment has three parts and each part can be performed by a group of 2-3 students.

Part 1 is the analysis of ethylene-vinyl acetate (EVAC) copolymers and the objective for this part is to record and analyze FTIR data of commercial EVAC random copolymers of different compositions. Set up a correlation graph of the absorbance ratio of specific IR peaks of different grades of EVAC copolymers and the PVAC content by elemental analysis. Finally based on the established correlation curves determine the per cent content of PVAC in an unknown sample of EVAC.

Part 2 is similar to part 1 except that a series of commercial PVP-PVAc copolymers will be studied.

Part 3 is similar to part 2 except that a series of instructor prepared PVP-PVAc blends will be studied.

### 2.1. Materials

The instrument used in this study was a Bruker Alpha-P® FTIR spectrometer having a Platinum® attenuated total internal reflectance (ATR) quick snap module containing a diamond crystal. The Alpha-P® unit has a Michelson Interferometer having a SiC globar as the IR source and the time dependence of the IR intensity is measured with a pyroelectric DTGS detector. The data was analyzed using the Bruker OPUS® software version 7.0.122. The spectra were displayed as absorbance vs frequency in wavenumbers ranging from 500 to 4000cm$^{-1}$. All of the Ethylene-Vinyl Acetate copolymers (EVAC) were obtained as solids in the form of pellets from Scientific Polymer Products (SP$^2$) Inc., Ontario, N.Y. The EVAC copolymers were randomized copolymers ranging from 9 to 70% PVAc. The Poly n-vinyl pyrrolidone - polyvinyl Acetate (PVP-PVAc) copolymers were also obtained from SP$^2$. These copolymers were also randomized copolymers ranging from 30 to 70% PVAc. The 30, 50, and 70% PVP-PVAc copolymers were obtained as 50 w/w% solutions in isopropanol. The PVP and PVAc homopolymers were also obtained from SP$^2$. Blends of the PVP and PVAc homopolymers were prepared by dissolving a PVP homopolymer having a Mw of 40K daltons with a homopolymer of PVAc having a Mw of 39K daltons in methanol (reagent grade) . These solutions were evaporated in a vacuum oven at 50$^0$C at 20 mm of Hg leaving solid blends of these homopolymers ranging from 20 to 80% PVAc. The 30%, 50% and 70% solid PVP-PVAc copolymers as well as the PVP-PVAc blends were all previous prepared by the instructor.

### 2.2. Methods

All the samples were analyzed with no sample preparation other than being pressed between the anvil and the diamond crystal from the ATR unit. They were all run as solids in which films were formed after compression. The samples were measured using 24 scans at a resolution of 4 cm$^{-1}$. Establishing the VA content is done using the ratio of absorbance of the carbonyl or C-O peak from the acetate of PVAc to either the C-H peak from PE or the carbonyl peak from the PVP . Elemental Analysis (EA) of the EVAC and PVP - PVAc copolymers were performed by the Galbraith Laboratories, Knoxville, TN. The %C & %H were obtained for the EVAC copolymers whereas the %C, %H, %N were obtained for the PVP - PVAc copolymers.

### 2.3. Hazards

There are no major safety issues for this experiment. All of the samples are commercial materials that are in solid form. Safety glasses are mandatory and the use of hoods is advisable. All waste materials are collected for waste disposal according to EPA and local guidelines.

### 2.4. Statistical Analysis

Each sample was analyzed multiple times. The data reported is an average of the normalized integrated peak areas obtained. The average percent relative standard deviation for the samples analyzed ranged from 0.3% - 1.3%. The elemental analysis of the % carbon, hydrogen, and nitrogen were reported as averages from duplicate tests.

## 3. Results and Discussion

Figure 1 through 5 show the ATR-FTIR spectra of: polyethylene, polyvinyl acetate, poly n-vinyl pyrrolidone, ethylene vinyl acetate copolymer having 33% PVAC, and a copolymer of PVP and PVAc having a PVAc content of

50%, respectively. Figure 1 shows the characteristics absorbance bands for PE which are located at: 2914 cm$^{-1}$, 2847 cm$^{-1}$, 1470 cm$^{-1}$ & 718 cm$^{-1}$. The 1470 cm$^{-1}$ & 718 cm$^{-1}$ peaks are used to identify and quantify the presence of PE. Figure 2 shows the FTIR of PVAc which exhibits a strong acetate ester absorbance band at 1728 cm$^{-1}$ due to the carbonyl group. Other specific absorbance bands for the PVAc molecule are: 1433 cm$^{-1}$, 1370 cm$^{-1}$, 1224 cm$^{-1}$ and 1016 cm$^{-1}$. The 1224 cm$^{-1}$ and 1016 cm$^{-1}$ are used to quantify and identify the presence of PVAc. Figure 3 shows that the major characteristic PVP absorbance peak is the 1650 cm$^{-1}$ which is from the carbonyl group. Figure 4 & Figure 5 show the major absorbance peaks present in the EVAC and the PVP-PVAc copolymer spectra, respectively.
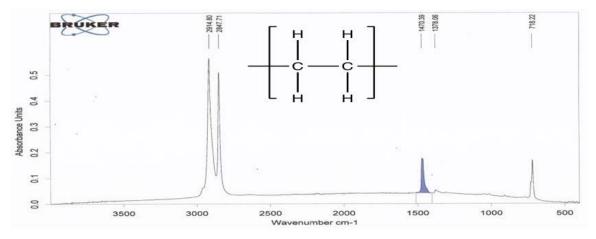


**Figure 1.** The FTIR spectrum of polyethylene (PE). The characteristic PE absorbance bands are located at 2914 cm$^{-1}$, 2847 cm$^{-1}$, 1470 cm$^{-1}$ (blue shaded peak), and 718cm$^{-1}$. The underline absorbance bands are used to identify & quantify the presence of PE
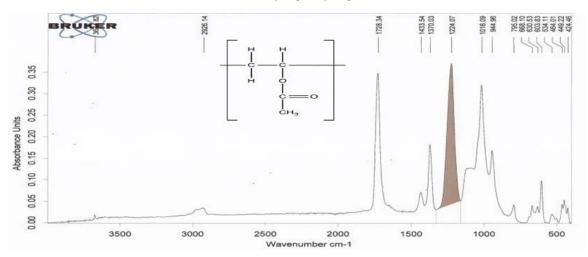


**Figure 2.** The FTIR spectrum of Polyvinyl Acetate (PVAc). The PVAc absorbance exhibited strong acetate ester carbonyl bands at 1728 cm$^{-1}$ (brown shaded peak). Other peaks are: 1433 cm$^{-1}$, 1370cm$^{-1}$, 1224 cm$^{-1}$, and 1016 cm$^{-1}$. The underline absorbance bands are used to identify & quantify the presence of PVAc
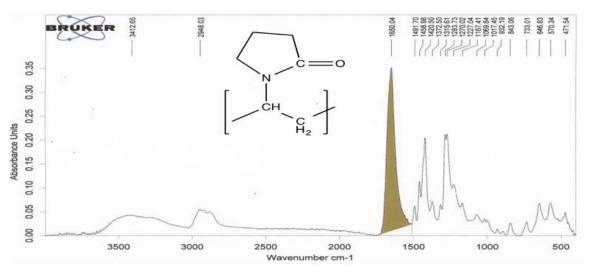


**Figure 3.** The FTIR spectrum of poly n-vinyl pyrrolidione (PVP). The major characteristic PVP absorbance band was 1650 cm$^{-1}$ brown shaded peak)
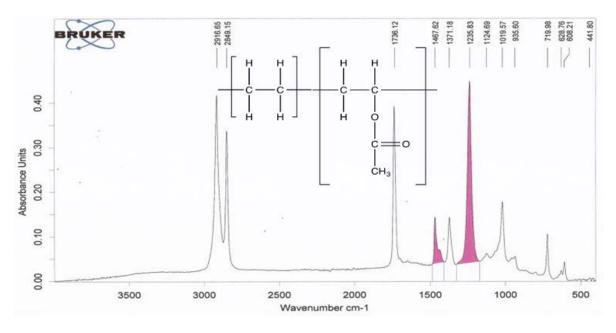
**Figure 4.** The FTIR spectrum of polyethylene-polyvinyl acetate copolymer (EVAc) having a 33% vinyl acetate content
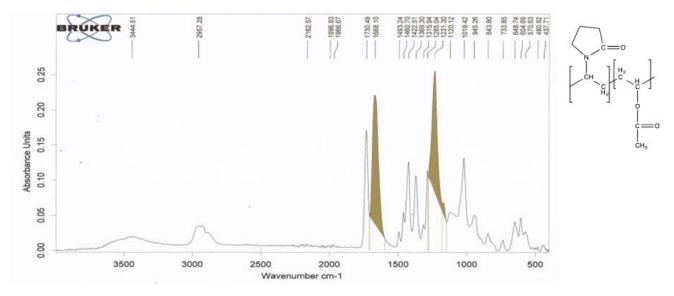


**Figure 5.** The FTIR spectrum of poly n-vinyl pyrrolidone –polyvinyl acetate copolymer (PVP-PVAc) having a 50% vinyl acetate content

## 3.1. PART 1: Experimental Study for the Ethylene – Vinyl Acetate (EVAc) Copolymers

### 3.1.1. Results and Discussion

Summarized in Table 1 below is the elemental analysis of the % carbon and % hydrogen for a series of EVAC copolymers having different amounts of poly vinyl acetate content ranging from 9 to 70% PVAc. The results show that as the % PVAc increases in the EVAC copolymer, the % carbon and hydrogen decreases and the % oxygen increases. The % oxygen was determined by difference between the sum of the % carbon and hydrogen and 100. Based on the experimental % oxygen the experimental % PVAc was calculated using equation 1:

$$\text{Exp \% PVAc} = \left[ \text{Exp \% Oxygen} \div 37.2 \right] \times 100. \quad (1)$$

Figure 6 is a plot of experimental % PVAc content vs the nominal % (as reported by the supplier) of PVAc provided by SP$^2$ shows a straight line relationship having a correlation coefficient of .999. This shows that based on the reported amount of PVAc in the EVAc copolymers from the supplier and the experimental determined amount of PVAc from elemental analysis (EA) corresponds exactly.
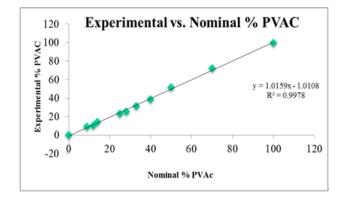
Table 2 summarizes the FTIR peak area ratios from specific absorbance peaks evaluated from the EVAC spectra. The table shows the peak area ratios of the 1236/1467 cm$^{-1}$ peaks, the 1020/720 cm$^{-1}$ ratio, and the 1735/720 cm$^{-1}$ ratio. In all cases as the % PVAc increases in the EVAC copolymers the ratios also increase. Figure 7, Figure 8, & Figure 9 show plots of the peak absorbance ratios of the 1236/1467 cm$^{-1}$ peaks, 1735/720 cm$^{-1}$ peaks, and the 1020/720 cm$^{-1}$ peak area ratios vs the experimental % PVAc present in the EVAC copolymers. All show straight line relationships however the best linear correspondence is shown in Figure 7 for the EVAC peak area ratio from the 1236 cm$^{-1}$ and 1467 cm$^{-1}$ vs the % PVAc. The correlation coefficient obtained from Figure 7 was .987.

**Table 1. A summary of the Elemental analysis of the % carbon and % hydrogen from a series of EVAC copolymers having a different amounts of vinyl acetate content**

| % PVAc in EVAc Copolymers (Nominal) | % Carbon | % Hydrogen | % Oxygen by difference (Note 1) | % PVAc experimental |
|---|---|---|---|---|
| 9 | 82.89 | 13.70 | 3.41 | 9.17 |
| 12 | 82.24 | 13.85 | 3.91 | 10.51 |
| 14 | 81.06 | 13.66 | 5.28 | 14.19 |
| 25 | 78.57 | 12.78 | 8.65 | 23.25 |
| 28 | 77.89 | 12.70 | 9.41 | 25.30 |
| 33 | 76.19 | 12.11 | 11.70 | 31.45 |
| 40 | 73.79 | 11.86 | 14.35 | 38.58 |
| 50 | 70.18 | 10.65 | 19.17 | 51.53 |
| 70 | 63.64 | 9.486 | 26.87 | 72.24 |
| 100 | 55.57 | 7.34 | 37.09 | 99.70 |
| Unknown-EVAc-1A | 81.07 | 13.80 | 5.13 | 13.79 |
| Unknown-EVAc-2A | 76.19 | 12.11 | 11.70 | 31.45 |

Note1: the % oxygen was determined by difference and the % PVAC was calculated using eq.1.
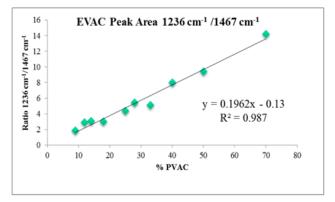


**Figure 6.** A plot of the experimental % PVAc vs the nominal % of PVAc provided by SP[2]



**Figure 7.** A plot of the EVAc peak area ratio from the 1236 cm$^{-1}$ & 1467cm$^{-1}$ vs the % PVAc

**Table 2. Summary table of the FTIR peak area ratios from specific peaks from EVAC copolymers**

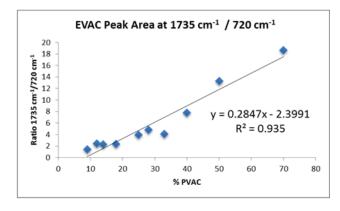| PVAc (nominal) | Peak Area Absorbance Ratio 1236cm$^{-1}$/ 1467cm$^{-1}$ | Peak Area Absorbance Ratio 1020cm$^{-1}$/720cm$^{-1}$ | Peak Area Absorbance Ratio 1735cm$^{-1}$/720cm$^{-1}$ |
|---|---|---|---|
| 9 | 1.85 | 1.01 | 1.45 |
| 12 | 2.91 | 1.79 | 2.45 |
| 14 | 3.05 | 1.85 | 2.27 |
| 25 | 4.39 | 2.82 | 3.98 |
| 28 | 5.45 | 3.77 | 4.79 |
| 33 | 5.12 | 3.40 | 4.10 |
| 40 | 7.98 | 6.82 | 7.81 |
| 50 | 9.39 | 18.19 | 13.27 |
| 70 | 14.21 | 18.61 | 18.67 |



**Figure 8.** A plot of the EVAc peak area ratio from the 1735 cm$^{-1}$ & 720 cm$^{-1}$ vs the % PVAc
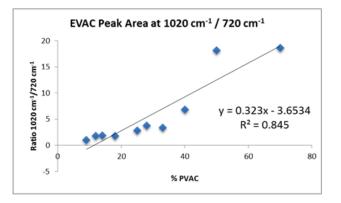


**Figure 9.** A plot of the EVAc peak area ratio from the 1020 cm$^{-1}$ & 720 cm$^{-1}$ vs the % PVAc

## 3.2 PART 2: Experimental Study for the PVP/PVAc Copolymers

### 3.2.1. Results and Discussion

Summarized in Table 3 is the elemental analysis of the % carbon, % hydrogen and % nitrogen of a series of PVP/PVAc copolymers having different amounts of PVAc content ranging from 30 to 70 % PVAc. The data show that as the % carbon, hydrogen and nitrogen increases, the % PVAc decreases. Based on the experimental % nitrogen determined, the % PVP was determined using equation 2 below

$$Exp\%PVP = [Exp\%nitrogen \div 12.61] \times 100. \quad (2)$$

The experimental % PVAc was determined by subtracting the % PVP from 100 as shown in Table 3. A plot, as shown in Figure 10, of the experimental % PVP vs the nominal % of PVP in the PVP-PVAc copolymer reported by the supplier shows a straight line relationship having a correlation coefficient of .986.

Table 4 is a summary of the FTIR peak area ratios for specific absorbance peaks evaluated from the PVP-PVAc spectra. Table 4 shows the peak area ratio from the 1667cm$^{-1}$ from the carbonyl group from the PVP and the 1231cm$^{-1}$ peak from the ether peak for the PVAc component. The table also shows the peak area ratios from the 1667cm$^{-1}$ for the carbonyl peak from the PVP and the 1735cm$^{-1}$ peak from the carbonyl group for the PVAc component. In both these cases as the peak ratios increase the % PVAc decreases. A plot of each of these ratios vs weight % PVP are shown in Figure 11 & Figure 12, respectively. The plot that gives the best correlation coefficient is from Figure 11. The correlation coefficient was .98 from the area absorbance ratio of the 1667 cm$^{-1}$ from the carbonyl group from the PVP component and the 1231cm$^{-1}$ peak from the ether group from the PVAc component.

**Table 3. A summary of the elemental analysis of the % carbon, % hydrogen, & % nitrogen of a series pf PVP/PVAc copolymers having different amounts of PVAc content**

| PVP-PVAc % (Nom) | % C | % H | % N | % O | Exp.% PVP (note1) | Exp.% PVAc (note1) |
|---|---|---|---|---|---|---|
| 30/70 | 58.31 | 7.450 | 4.379 | 29.86 | 34.73 | 65.27 |
| 50/50 | 59.05 | 7.743 | 6.239 | 26.91 | 49.48 | 50.52 |
| 60/40 | 58.50 | 7.670 | 6.959 | 26.87 | 55.19 | 44.81 |
| 70/30 | 60.37 | 8.083 | 7.659 | 23.89 | 60.74 | 39.26 |

Note 1: Based on the % nitrogen, the % of PVP was determined and by difference the % PVAc was obtained. Elemental analysis was done by Galbraith Labs, Knoxville, TN.
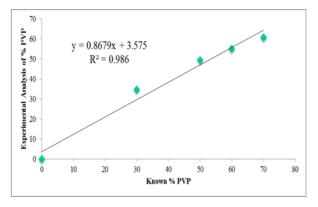


**Figure 10.** A plot of the experimental % PVP vs the nominal % of PVP in the PVP-PVAc Copolymer

**Table 4. A summary of the FTIR peak area ratios for the specific absorbance peaks from the PVP-PVAC copolymer spectra**

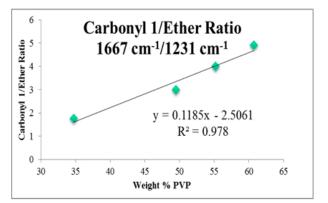| Experimental Wt % PVP/PVAc | Ratio Carbonyl 1/Ether peaks 1667cm$^{-1}$/1231cm$^{-1}$ PVP / PVAc | Ratio Carbonyl 1/Carbonyl 2 1667cm$^{-1}$/1735cm$^{-1}$ PVP / PVAc |
|---|---|---|
| 34.7/65.3 | 1.77 | 1.92 |
| 49.5/50.5 | 2.99 | 2.71 |
| 55.2/44.8 | 4.00 | 3.63 |
| 60.7/39.3 | 4.92 | 5.39 |



**Figure 11.** A plot of the weight % PVP vs the ratio of the peak area absorbance form the 1667 cm$^{-1}$ peak from the carbonyl group on the PVP and the 1231 cm$^{-1}$ peak from the C-O peak from the PVAc
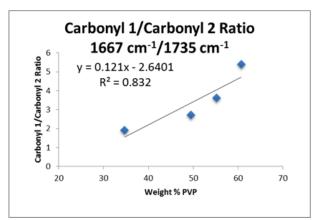


**Figure 12.** A plot of the weight % PVP vs the ratio of the peak area absorbance form the 1667 cm$^{-1}$ peak from the carbonyl group on the PVP and the 1735 cm$^{-1}$ peak from the carbonyl peak from the PVAc

## 3.3. Part 3: Experimental Study for the PVP – PVAc Blends

### 3.3.1. Results and Discussion

Table 5 summarizes the data from our experimental study for the PVP – PVAc blends which show that as the % PVAc increases in the prepared blends the ratio of the carbonyl absorbance peak from the PVP and the 1231cm$^{-1}$ peak from the ether group from the PVAc decreases proportionally as shown in Figure 13.

**Table 5. A summary of the data of the weight % of PVAc in the prepared PVP – PVAc blends versus the peak area ratios of the carbonyl peak form the PVP and the ether peak from the PVAc**

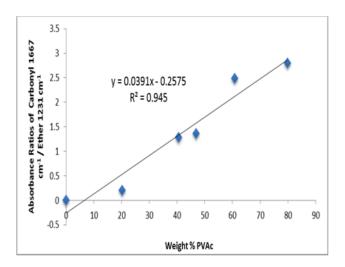| Wt % PVAc | Ratio of Carbonyl 1 / Ether Peaks 1667 cm$^{-1}$ /1231 cm$^{-1}$ |
|---|---|
| 20.17 | 2.8 |
| 39.26 | 2.49 |
| 43.09 | 1.365 |
| 59.50 | 1.29 |
| 79.78 | 0.2068 |

**Figure 13.** A plot of the absorbance ratios of the carbonyl 1667 cm-1 peak for the PVP component and the ether 1231 cm -1 peak from the PVAc component versus the weight % of PVAc

## 4. Conclusions

FTIR and elemental analysis can be used to quantify the amount of PVAc in copolymers of ethylene and PVP and blends prepared with PVP. The experiments described in this report demonstrated that simple, nontoxic and nondestructive ATR absorbance measurements can be made to quantitatively evaluate the polymer contents in simple commercial copolymers. Peak area ratios at signature signals that correspond to key vibrations can be used to determine the weight percent of a polymer component in a binary polymer blends. The FTIR procedure and quantitative method is simple and can be readily adopted for undergraduate quantitative or instrumental analysis lab. The students can also utilize these experiments to enhance their understanding of polymer compositions and related vibrational patterns that can be detected by FTIR spectroscopy.

## Acknowledgements

## Statement of Competing Interests

The Authors have no competing interests.

## List of Abbreviations

All the abbreviations are defined in the body of the text.

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