Effective Integration of Molecular Spectroscopy in Undergraduate Research Experiences

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Introduction

STEM research has proven to be beneficial in students learning, retention and graduation rates, entry into to the graduate and professional programs, and development of the critical thinking and soft skills. Keeping this in mind, over the last several years we have carried out a wide variety of molecular spectroscopy undergraduate projects in areas of biomedical studies [1], hydrogen bonding [2], pharmaceutical research [3] and blended fuels assessment. Three topics from our research are exemplified in this poster.

Experimental

FT-IR spectra were recorded on a Bruker Vector 33 spectrometer equipped with a KBr beam splitter and deuterated triglycine sulfate (DTGS) room temperature detector. The spectra were recorded in the attenuated total reflection (ATR) mode with a single bounce MIRacle^R ATR accessory from PIKE technologies, INC. (Madison, WI). The accessory was equipped with a split pea shaped ZnSe ATR crystal. The near infrared spectra (NIR) were recorded on the same instrument as the FT-IR spectra in the absorbance mode using 1 mm rectangular quartz cell. The NIR spectra were recorded by using Tungsten sources, CaF_2 beam splitter and Ge detector.

Projects

Hydrogen Bonding: We investigated the hydrogen bonding in isopropanol-water and t-butanol-water mixtures in the mole fraction range 0.1 to 0.9 through the application of infrared spectroscopic technique.

Blended Fuels: (NIR) spectra of several ethanol blended gasoline samples collected from the different gas stations in the Palm Beach County of the State of Florida (USA). The ethanol content of the gas samples was predicted from the previously established NIR calibration in our laboratory.

Pharmaceutical Study: Contamination of cough syrups with Diethylene Glycol (DEG) was studied using both near-iinfrared and infrared techniques.

Results

Hydrogen Bonding: Figure 1 displays infrared spectra of isopropanol water mixtures with mole fraction of 0.1-0.9. The spectra of t-butanol and water mixtures were also studied but are not shown. The integrated areas of the C-O stretching band (~1000 cm^{-1}) versus mole fractions were plotted for both mixtures and are shown in Fig. 2. The C-O plot areas were used to explain the molecular associations in the mixtures studied. These results indicate two nonequivalent hydrogen bonding clathrates in the mixtures.

Blended Fuels: The major peaks of the spectra (Figure 3) are due to CH stretching first overtone (~5670, ~5770, ~5810 and ~5870 cm^{-1}), CH stretching second overtone (~8285, ~8400 and ~8690 cm^{-1}) and combinations peaks (~7500-6500 cm^{-1}). The predicted ethanol percentage in the gasoline samples was in the range of 4.34-6.68 instead of generally assumed value of 10 percent.

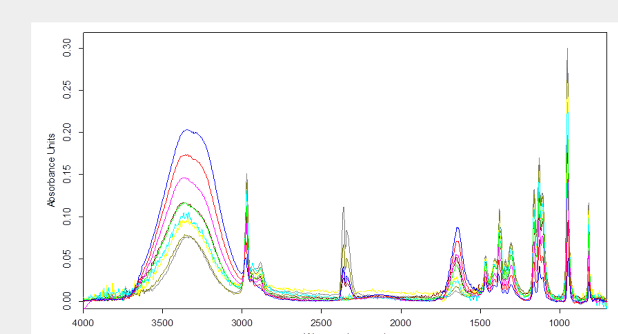

FIGURE 1 FT-IR spectra of isopropanol-water mixtures in the mole fraction range 0.1 to 0.9.

Figure 1. Infrared spectra of isopropanol water mixtures in the 0.1 to0.9 mole fraction range.

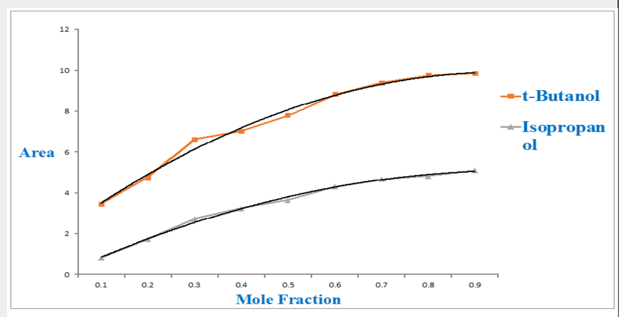

FIGURE 5 Integrated area plots of C-O stretching bands of isopropanol-water and t-butanol-water mixtures in the mole fraction range 0.1 to 0.9.

Figure 2. Plots of integrated areas of C-O stretching bands of isopropanol-water and t-butanol-water mixtures in the mole fraction range of 01-0.9.

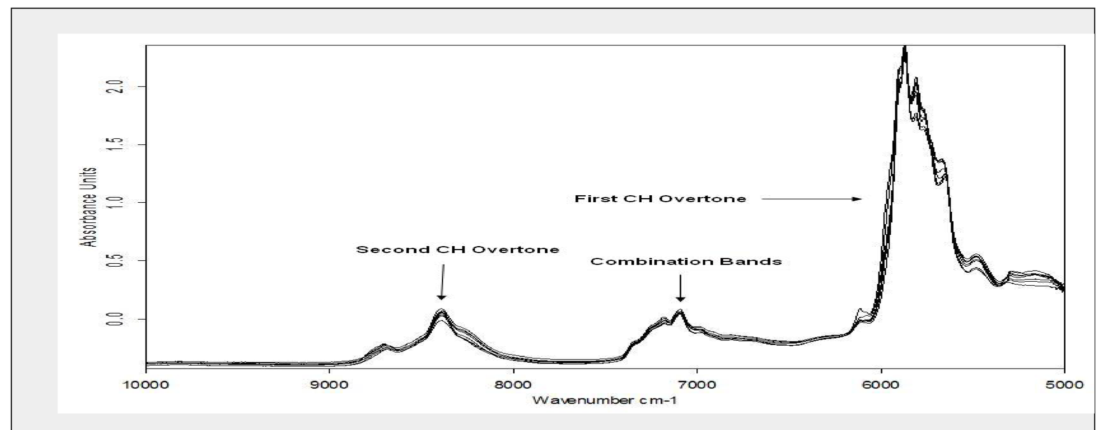

Figure 3. Near infrared spectra of E-10 gasoline samples from different fuel stations in Palm Beach County.

Further research is needed to ascertain the reason for this disparity. Cost presents a possible cause. Although E-10 is said to be a cheaper alternative, ethanol is generally more expensive to produce than gasoline.

Pharmaceutical Study: The consumption of cough syrups contaminated with Diethylene Glycol (DEG) has devastating effects on the health. Fourier transform infrared (FT-IR) and near infrared(NIR) spectroscopic techniques are viable, simple, cost effective, rapid and fool proof methods for the identification and quantification of DEG in glycerin based cough syrups. The FT-IR and NIR spectra of the glycerin based cough syrup and up to 50:50 mixtures of DEG in cough syrup are recorded. The major peaks in the FT-IR spectrum of the cough syrup are assigned to the OH stretching (~3300 cm^{-1}), CH stretching (~2900 cm^{-1}), CH bending (1500-1200 cm-1) and C-O stretching (1200-900 cm-1) vibrational modes.

In the FT-IR spectra of the mixtures, DEG contribute distinct peaks (Figure 4) due to the vibrations of the C-O (920 cm^{-1}) and OC_2H_4 (892 cm^{-1}) moieties of its backbone and form the basis of the DEG detection and quantification. The prominent peaks of the NIR spectra (Figure 5) of cough syrup and DEG are assigned to the first overtones of OH and CH, and to the combination of OH and CH fundamental vibrations. Both FT-IR and NIR spectra predicated the amount of DEG contaminant within 1% of the actual value which shows that both of these techniques can be used to screen DEG contaminated cough syrups.

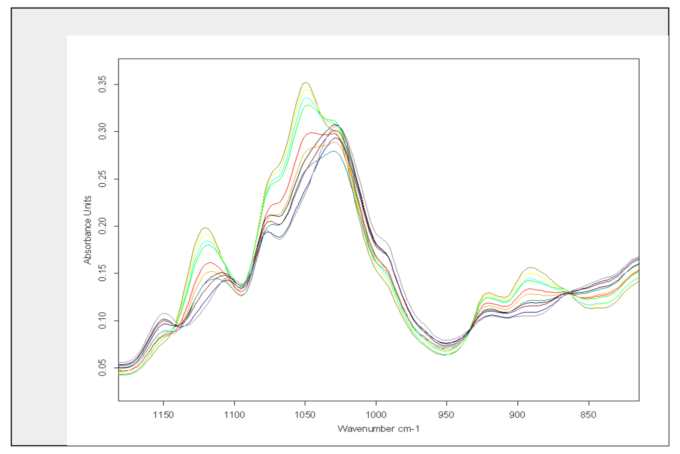

Figure 4. Infrared spectra (1200-800 cm^{-1}) of cough syrup contaminated with up to 50 percent diethylene glycol contaminant.

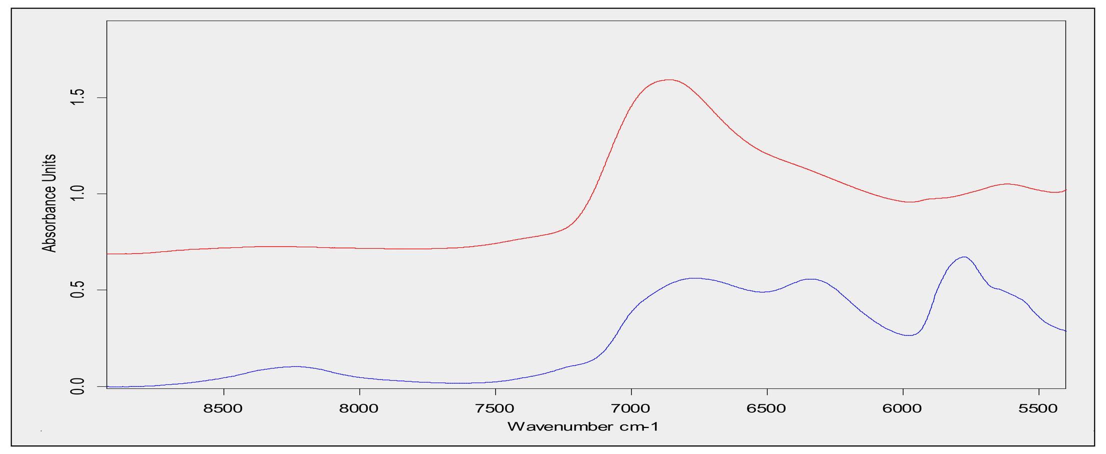

Figure 5. Near Infrared spectra of cough syrup (red) and diethylene glycol (blue).

References

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Conclusion

Molecular spectroscopy which is an important physical technique can easily be incorporated in the undergraduate research programs to provide useful hands on research experience to the students. Such experience is mutually beneficial to the faculty, students and the institution. At four years colleges where faculty has heavy teaching load, involvement of undergraduates in research can significantly lead to higher research output of the faculty. Moreover, such experiences can lead to success for students and can also be counted as professional development for the faculty.

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