

Raman Shift Spectroscopy

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ABSTRACT

The primary objective of this experiment is to understand the method of Raman Shift spectroscopy by calculating the Raman shift of five well known samples: Corn Starch, Calcium Sulfate, Deionized (DI) Water, Methanol and 1:1 part water-methanol mixture. Our results for Methanol, Calcium Sulfate and Cornstarch were notably consistent with our researched spectra.

Raman Spectroscopy

1 Introduction

Considering all matter is composed of molecules that are never in stasis (unless they are at absolute zero), we can exploit the vibrational properties of molecules that rely on the associated quantum energy levels to create distinctive spectra for identification of substances. C.V. Raman discovered Raman Scattering in 1928 and was later awarded the Nobel prize for his discovery. Raman Shift spectroscopy relies on the scattering of an incoming monochromatic light source on a sample and the reflected light beam that returns to the equipment light source detector. The monochromatic light source interacts with molecular vibrations or other excitation in the system, resulting in the energy of the photons being shifted up or down. This shift in energy gives information about the vibrational modes of the system.

2 Experimental Setup

To begin, we check the equipment's connections as well as the power supply. Next, we open the "Spectra Suite" software, and ensure that "Data Sources" is adjusted to "MMS-Raman" instrument. After that, we select "Processing" → "X axis units" → "Raman shifts," and change the "intergration time" between 1 and 5 seconds.

In order to obtain clean and consistent Raman signals, different parameters are adjusted, such as the integration time (absolute signal) and "scans to average." To begin the physical process, we load an empty sample cuvette to collect the "background noise" (or spectra which we will subtract from all of the spectra we want to analyze). To acquire spectra plot, we choose "Spectrometer" → "Acquisition." Then, the plot of the spectrum is autoscaled by using the "up/down arrow icon" in the graph toolbar will autoscale. We also adjust the positioning knob on the sample mount to achieve a maximum signal for the loaded sample. When we are satisfied with our result (when the spectra is properly consistent with our research Raman shift spectra), the collected data is saved and later processed. We repeat the above process with five different samples: DI water, 1:1 ratio of DI water and Methanol, CaSO_4 , Corn starch and Methanol.

3 Result

Figure 1 below displays the Raman shift for DI water sample that we obtained from "ResearchGate" to compare to our result (See Figure 2). After subtracting the background noise (empty cuvette spectrum) from our collected data for DI water, we were able to generate a fairly precise Raman shift for our sample that bears a notable resemblance to the spectral sample from "Research Gate". Comparing the two figures (1 and 2), the Raman shift is very similar for the range of 500-1500, however, the intensity of 2 plots are different. One possible cause of inconsistency could be minute variations in the wavelength of laser sources used. Although the collected spectrum seems fairly plausible, we naturally expect a few inevitable errors. Another cause of inconsistency could be external vibrations from the surrounding environment disturbing the Raman Shift equipment. As we know, Raman Shift Spectroscopy is a spectroscopy technique that relies on detecting nearly atomic sized variations of molecular vibrations, it is an extremely sensitive spectroscopy technique. Therefore, error resulting from even the most minuscule vibrations is practically inevitable in our university laboratory environment.

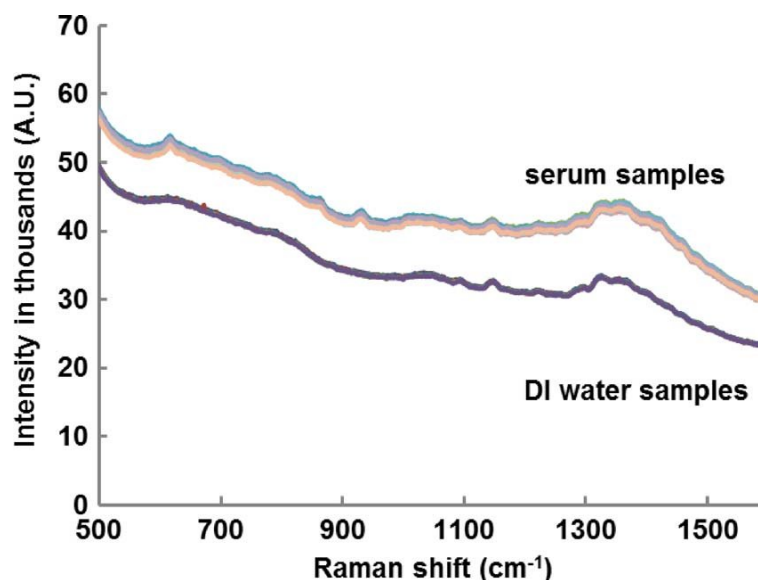


Figure 1. Raman spectra of DI water¹.

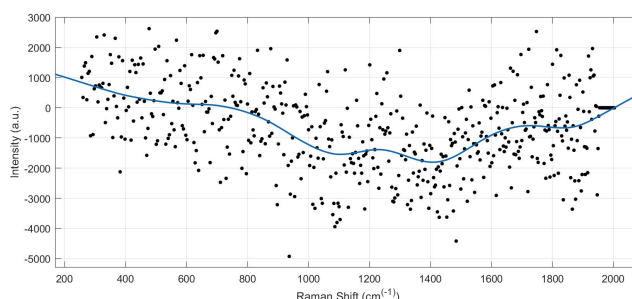


Figure 2. Collected Raman spectra of DI water after subtracting the background noise.

Similarly, we obtained a Raman shift spectrum from "ResearchGate" for methanol in water with 1:1 ratio (See Figure 3). After subtracting the background noise (empty cuvette spectrum), we were able to generate a Raman shift for our samples that have similar notable features to the spectral sample from "Research Gate". Looking at Figure 4, we obtained a peak roughly around 1010 cm^{-1} . Furthermore, we also noticed a similar peak at the same position in the sample spectrum (Figure 3). Thus, we can assume that our collected data is accurate, and the variation of intensity can potentially be the result of different laser sources. Again, we expect the same percentage of error that comes from the surrounding's vibration.

Moreover, by comparing Figure 5, from "Characterization of Starch by Vibrational Spectroscopy" by Brandon Holder, and 6, we recognized similarly peaks at almost the same positions between the two figures (Figure 5 and 6). For example, we obtained a peak at 490 cm^{-1} in both figures, and another one at roughly 1450 cm^{-1} . However, there is a significant peak in our collected spectra at 950 cm^{-1} (See Figure 6). Analyzing the same area there in Figure 5, we can see a small peak, which is not as prominent as our spectra. Again, the variation in intensity is poignant. As we mentioned above, the intensity's variation can be the result of different light sources. However, it is possible that our samples were contaminated. In order to verify this, we would need to run at least 5-10 additional trials with new samples and compare the results. Although there are some different between the two figures (Figures 5 and 6), we determine that our spectra has a notable resemblance to the sample's model. Hence, we've come to

the same general conclusion, Figure 6 is one of our best results.

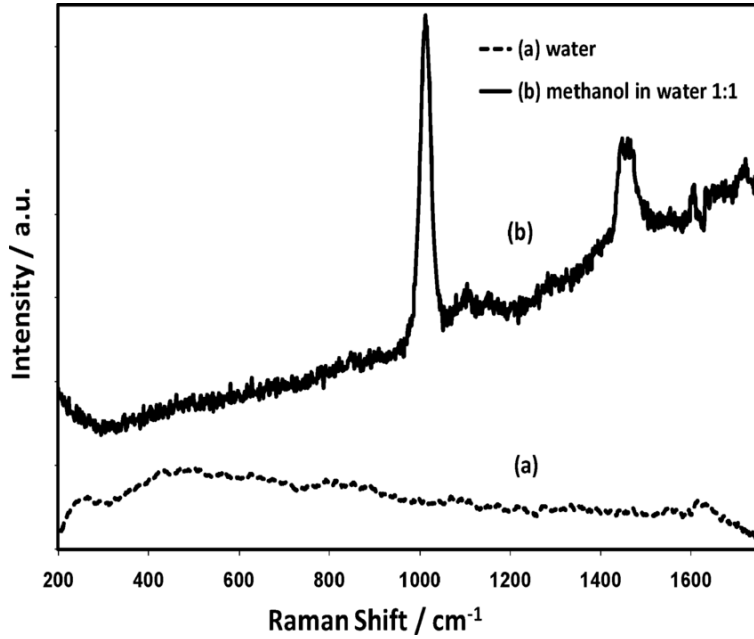


Figure 3. Raman spectra of liquids in clear plastic cuvette: (a) water and (b) methanol in water, 1:1 mixture².

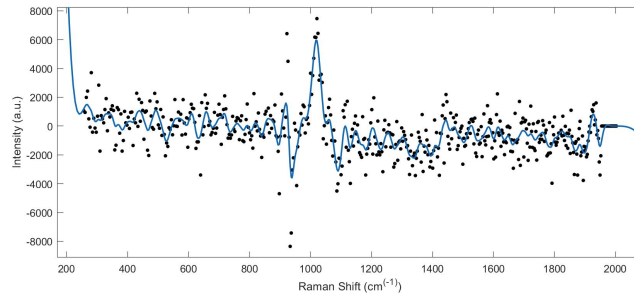


Figure 4. Collected Raman spectra of methanol in water (1:1) after subtracting the background noise.

Additionally, we acquired a Raman shift spectra of CaSO_4 from “A miniaturized Raman/LIBS instrument for in-situ investigation of celestial bodies in pioneering missions” by Simon Kubitz and et al (See Figure 7). Looking at the two figures (Figures 7 and 8), we can see quite a drastic difference in intensity. In Figure 8, the intensity seems to decrease over larger shifts, however, the intensity in Figure 7 remains consistent. Again, our samples could potentially be contaminated, or the vibration from the surrounding equipment and human steps could have impacted our collected result. However, we were able to successfully detect the characteristic peak of SO_4 at 990 cm^{-1} , which is similar to the sample spectra. Hence, our collected spectra for CaSO_4 is far less than ideal, but it still allows us to observe the physical property of the substance.

For Methanol, our measurements produced excellent spectra (See Figure 10) because of the characteristic peaks. In the range of 200 cm^{-1} to 2000 cm^{-1} , we detected two characteristic peaks of Methanol at 1120 cm^{-1}

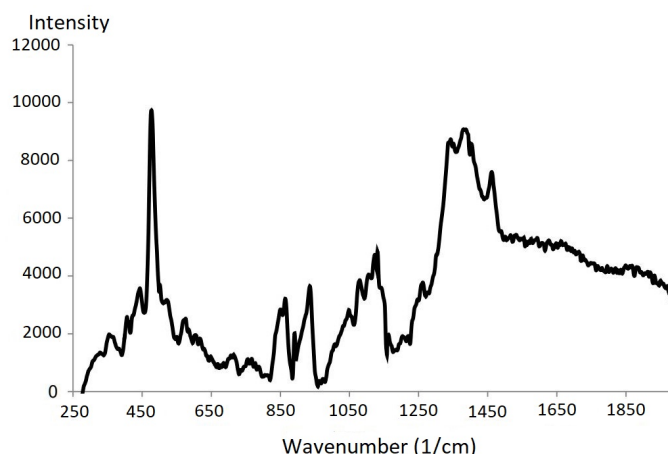


Figure 5. Raman spectrum of normal cornstarch collected using 785 nm (near-infrared) excitation³.

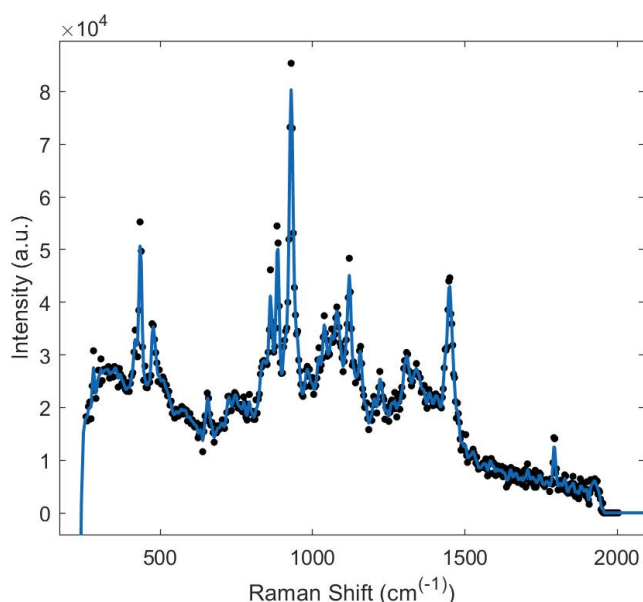


Figure 6. Collected Raman spectra of cornstarch after subtracting the background noise.

and at 1500 cm^{-1} . By comparing our result to Methanol spectra sample from “Quantitative Evaluation of Methanol Content in Beverages Based on Raman Spectral Data” by Hana Vaskova (See Figure 9), we determined that our collected result has an uncanny resemblance to the sample’s model. Thus, we can make an assumption that Methanol spectra is our best collected sample.

4 Conclusion

We realized that our collected results for aqueous samples were notably more stable than for non-aqueous samples, which were noisy by comparison. This is consistent with our assumptions of the general operation of the equipment as we collect data for each sample. As aforementioned, our most stable and reliable readings came from Methanol, CaSO_4 , and Cornstarch. We typically anticipate a few errors during our experiment procedure. In order to improve collected data, we believe that running multiple additional trials and replacing old samples will

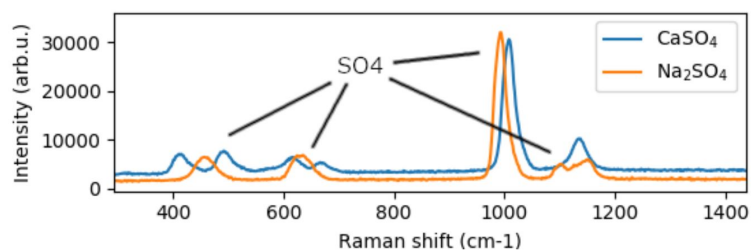


Figure 7. Raman spectra of CaSO_4 and Na_2SO_4 ⁴.

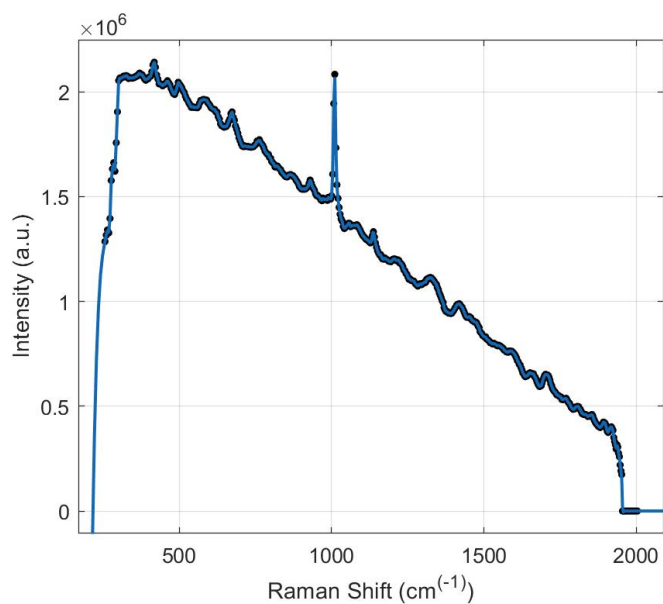


Figure 8. Collected Raman spectra of CaSO_4 after subtracting the background noise.

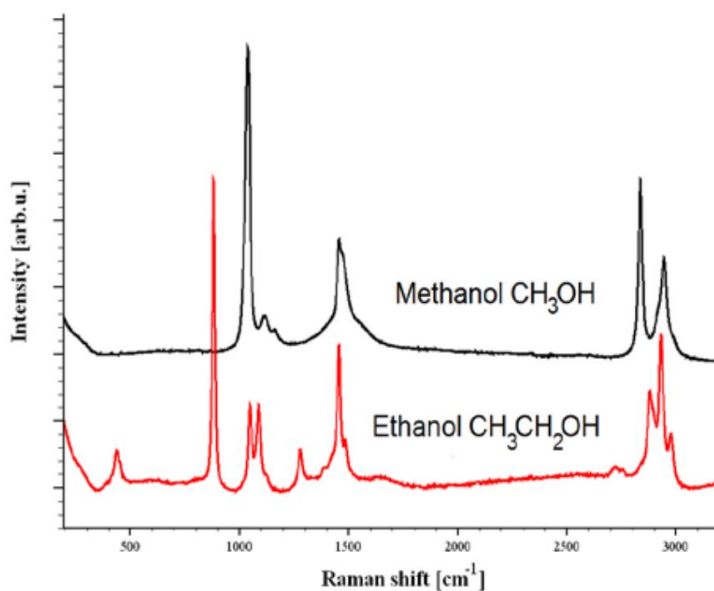


Figure 9. Raman spectra of Methanol and Ethanol⁵.

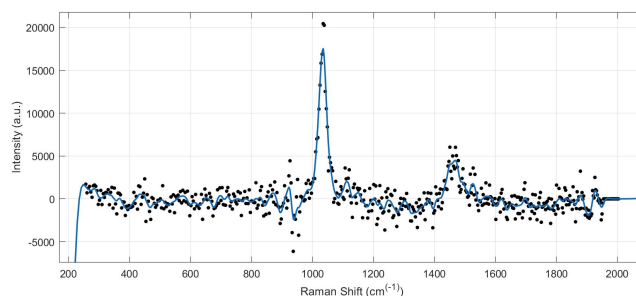


Figure 10. Collected Raman spectra of methanol after subtracting the background noise.

help to eliminate the error causing from sample contamination. As aforementioned, Raman Shift Spectroscopy is extremely sensitive to vibrations. Therefore, to obtain better spectra, we can potentially try to eliminate the surrounding vibration by moving the lab station to a more stable working environment with less vibrations from surrounding equipment and less human steps.

References

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