

How to Incorporate Technology with Inquiry- Based Learning to Enhance the Understanding of Chemical Composition; How to Analyze Unknown Samples

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ABSTRACT

The use of technology in teaching offers numerous amounts of possibilities and can be challenging for physics, chemistry and geology content courses. When incorporating technology into a science content lab it is better to be driven by pedagogy than by technology in an inquiry-based lab setting. Students need to be introduced to real-world technology in the beginning of first year chemistry or physics course to ensure real-world technology concepts while assisting with content such as periodic trends on the periodic table. This article will describe the use of technology with Raman Spectroscopy and Energy Dispersive X-Ray Spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FTIR) to research chemical compositions in the real world of unknown samples. Such unknown samples utilized in this lab were clamshell (parts of clams that look like shark teeth) versus shark teeth. The data will be shared to show how the students (pre-service teachers and in-service teachers) solved the problem using technology while learning important content that will assist in the next level of chemistry, physics and even geology.

INTRODUCTION

Teaching with technological instrumentation such as EDS, Raman Spectroscopy and FTIR in the first year of chemistry can deepen student learning and thus support instructional objectives. It is important to choose the best technology/instrumentation related to the content without losing sight of the goals. One of the challenges of teaching chemistry, physics and geology is making labs that are not cook book format but require students to find the answer on their own through discovery –based learning. This paper describes how students will build content knowledge in chemistry, physics and geology while utilizing

technology based instrumentation such as Raman Spectroscopy, FTIR, and Energy Dispersive X-Ray Spectroscopy analysis to solve the problem of what fossils may be their unknown sample given in the inquiry-based technology lab. The two instrumentation techniques used for the students to determine their unknown sample analysis has allowed their building of content knowledge with interdisciplinary science.

Raman spectroscopy named after Sir C.V. Raman is used to observe the vibrational, rotational and other low frequency modes. This is a nondestructive tool used to analyze a sample that is based upon inelastic scattering. Raman spectroscopy is typically used to give an overall fingerprint of molecules within the sample analyzed. A laser light from the Raman instrument interacts with the molecular vibrations or phonons. The Raman has light scattered off a sample as opposed to absorbed by the sample being analyzed. Also, Raman requires no sample preparation and is insensitive to aqueous absorption bands which facilitates the measurements of solids, liquids or gases directly. Raman spectroscopy can have fluorescence problems with the sample, but this can be overcome as will be illustrated in this article.

Energy Dispersive X-Ray Spectroscopy (EDS) is an analytical tool used for the elemental analysis of a sample. EDS instrumentation focuses beam source of X-rays onto the sample which provides an X-ray excitation. The X-ray emission spectrum that is produced from a sample (fossil sample) is due to the fundamental principle that each element has a unique atomic structure thus exclusive peaks on the X-ray spectrum for each sample analyzed. The sample is hit with the incident laser beam that could excite an electron in an inner shell where an electron could be ejected. The area where the electron is ejected creates a hole in the inner shell

that would be filled by an electron from the higher-energy shell. This causes an X-ray released due to the difference in energy as described above (the difference in energy from the higher energy shell to the lower energy shell). The Energy Dispersive Spectrometer measures the number and energy of the X-rays emitted from a sample. Therefore, the EDS allows for the measurement of the sample to be determined and compared based upon the principles of general chemistry and atomic structure.

The FTIR instrumentation deals with the infrared region of the electromagnetic spectrum and assists in identification of the chemical composition/chemical functional groups of the sample. The advantages of FTIR instrumentation are no fluorescence problems as associated with Raman spectroscopy.

EXPERIMENTAL

Apparatus

The sample of shark tooth was examined with a Nomadic Raman Microscope with two different lasers; 1064 nm and 785 nm to compare the analysis of the shark tooth with assistance from BaySpec. The detector was a TEC cooled InGaAs detector arrays with an Olympus Bx51 upright microscope and imaging camera with automated stage for mapping. The samples of clam and shark tooth were examined with a Bruker Quantax EDS with SEM which features the XFlash 6 detector series with active areas from 10 -100 mm². The generation 6 provides the hardware and software technology to deliver the fastest and most reliable results. Also, the older version of the Bruker SEM-EDS instrument was utilized as well with the assistance of Bowser-Morner. The Nicolet iS 5 FTIR Spectrometer by Thermo Scientific was utilized to analyze the samples.

RESULTS/DISCUSSION

The students were required to compare the two different types of fossils (shark tooth versus clam shell) and determine the chemical composition of each unknown fossil. Students were given a pre-test to predict what the chemical composition of a tooth versus a clamshell may exhibit related to instrumentation technology. The pre-test score was 30% for the understanding of chemical composition for these two different types of fossils and at the end of the lab they were given a post-test where there was a 100% gain; thus a net 70% gain in content was exhibited with the integration of Raman and EDS technology. The clamshell would be composed of calcium carbonate where a shark tooth would exhibit calcium phosphate. The instrumentation technology helps the students analyze the different samples and see the detailed difference between the chemical compositions of these two different fossils. Figure 1 illustrates the challenge encountered with Raman spectroscopy where samples can exhibit fluorescence and prevent the spectra from being exhibited with

prominent peaks. Figure 2 displays the Raman spectra of shark tooth analyzed with a different laser (1065 nm) that prevents the fluorescence from occurring (note the prominent peaks labeled in detail for the phosphates and carbonate). Thus, Figure 2 shows that the first three peaks are due to phosphate, and confirm the calcium phosphate chemical composition of the shark tooth. Unknown could not be a clamshell which would be composed of calcium carbonate. However, it is significant to note that two-three lasers are an expensive requirement to analyze a sample and prevent fluorescence. Therefore, EDS has shown to be the best technique to analyze the two samples and distinguish the shark tooth versus the clamshell without the costly use of several lasers. Figure 3, and Figure 4, illustrate how the EDS calculates the percent of each element present in the sample analyzed without any fluorescence issues. Figure 5 A, (FTIR) and 5B, (Raman), show how for the fossil materials it was better to utilize the FTIR than the Raman spectroscopy due to the effect of the autofluorescence issue as shown in Figure 5B (thus peaks not distinguished in Raman). Thus, in the Raman spectra Figure 5B, the peaks are not discerned as in the FTIR Figure 5A. The Raman can have autofluorescence and this can be due to the fact that geological samples/fossils have impurities due to transition metals such as chromium, vanadium, and manganese present.

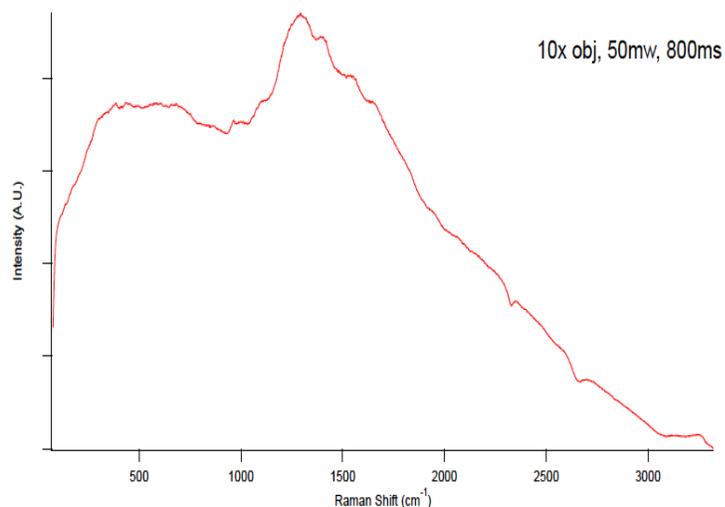


Figure 1. Raman spectra of shark tooth with 785 nm laser.

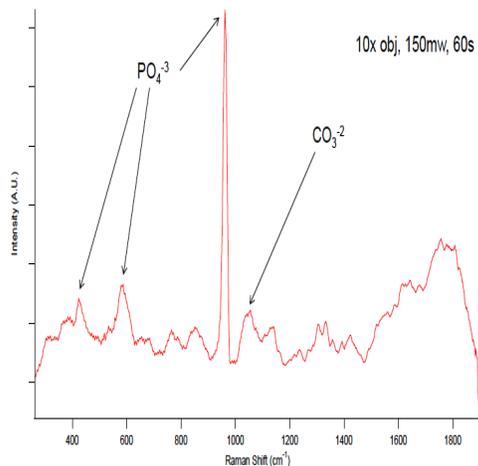


Figure 2. Raman spectra of shark tooth with 1064 nm laser.

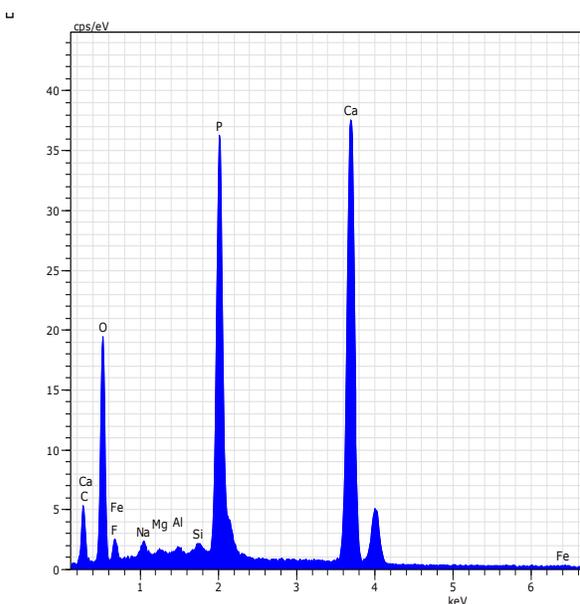


Figure 3. EDS of shark tooth with the following:

El AN Series	Net norm. C	[wt.%]
C 6 K-series	4369	12.42
O 8 K-series	19723	43.98
F 9 K-series	1842	4.19
Na 11 K-series	2254	1.20
Mg 12 K-series	1243	0.43
Al 13 K-series	1334	0.36
Si 14 K-series	1953	0.37
P 15 K-series	53731	11.71
Ca 20 K-series	77992	25.22
Fe 26 K-series	191	0.13

Total:		100.0

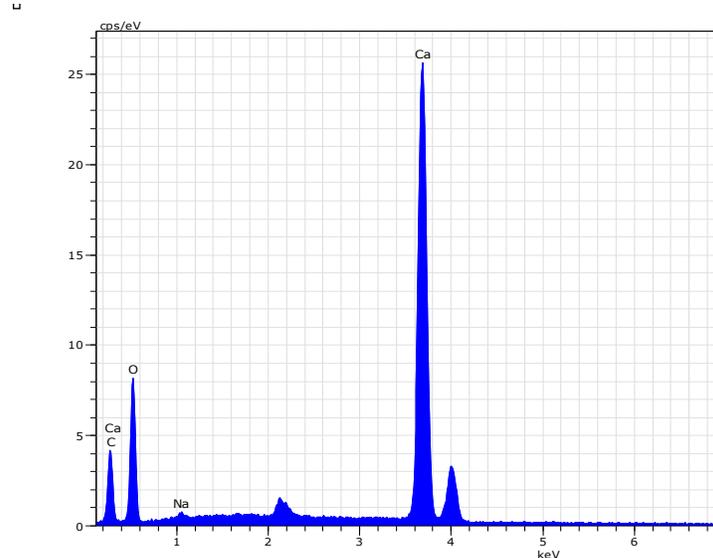


Figure 4. EDS of Clamshell with the following:

El AN Series	Net norm. C	[wt.%]
C 6 K-series	5676	11.48
O 8 K-series	14122	50.52
Na 11 K-series	661	0.39
Ca 20 K-series	92663	37.61

K-alpha 0.5
Beta 4ev Ca
Total: 100.00

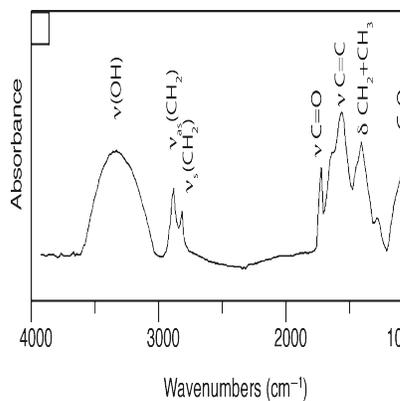


Figure 5A.

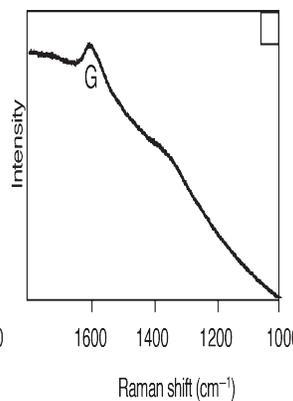


Figure 5B.

Figure 5A., FTIR of fossil with functional groups OH, CH₂, CO, C=C, CO and **Figure 5B.,** Raman spectra of fossil; Graph from Marshall and Marshall : Spectroscopy of Fossils

CONCLUSION

This inquiry-based experience with our students has assisted their content knowledge with novel technology and analytical instrumentation such as Raman spectroscopy, EDS, and FTIR that may be utilized in the chemistry, physics, geology and even forensics labs to classify the chemical composition of the unknown sample. The pre-test and post-test analysis has exhibited an increase in content knowledge as inquiry-based lab experiences have been cited by R.R. Hake' to promote learning. These experiences have been valuable to work with industry such as Bowser-Morner to build students' understanding of technology related to real-world analysis needed to solve real-world problems. Additionally, the pre-service and in-service teachers can better assist their own future students with inquiry-based learning experiences and collaborate with industrial settings such as Bowser-Morner.

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