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Research Article

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Raman Spectroscopy: A Proposal for Didactic Innovation (IKD Model) In the Experimental Science Subject of the 3rd Year of the Primary Education Degree

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Abstract
In recent years, new innovative teaching methodologies have been used in the Basque Country University (IKD model) for experimental science classes for teachers of Primary Education in formation. This IKD model is based on a cooperative and dynamic learning. It is an own, cooperative, multilingual and inclusive model that emphasizes that students are the owners of their learning and are formed in a comprehensive, flexible and adapted to the needs of society. Training students according to IKD model requires creating new ways of teaching and learning more active and cooperative (curriculum development). Therefore, the fact of combining more theoretical master classes with more practical classes is a trend that is increasingly used. For this reason, combining concepts of chemistry and physics assumes that the student relates different ideas that are a priori complex and unrelated, but in the scientific field, they are related. In this way, the work presented is a proposal of methodology for the subject of experimental sciences for teachers in training, which will introduce real scientific instrumentation such as Raman spectroscopy, which can be of great interest to perform significant learning and to design teaching-learning activities.

Keywords: Didactic Methods, IKD Model, Raman Spectroscopy, Crystallography, Laser

Introduction

Raman spectroscopy is a high-resolution photonic technique that provides in a few seconds chemical and structural information of almost any organic or inorganic material or compound, allowing also its identification. The analysis by means of Raman spectroscopy is based on the examination of light scattered by a material when a monochromatic (single color) beam of light hits it. A small portion of the light is dispersed inelastically undergoing slight changes in frequency that are characteristic of the material analyzed and independent of the frequency of the incident light. It is an analysis technique that is performed directly on the material to be analyzed without needing any special preparation and that does not entail any alteration of the surface on which the analysis is carried out, that is, it is non-destructive. This type of instrumentation allows to obtain in a very fast way all type of molecular information of any type of material. In addition to its easy and fast handling, it does not cause the destruction of the sample, which favors the increasingly wide use of Raman spectroscopy in gemology and mineralogy.

The phenomenon known as Raman Effect was discovered by the Indian physicist Chandrasekhara Venkata Raman, in the year 1928, which earned him the Nobel Prize in physics in 1930. This scientist gave name to the inelastic phenomenon of scattering of light that allows the study of rotations and molecular vibrations. In 1923, while studying the dispersion of light in water and in purified alcohols, one of his students observed a change in color in a ray of sunlight as it was filtered and traversed the material under study and his team could not eliminate this effect. Therefore, they suspected that the phenomenon was a characteristic property of the substance. After conducting several studies over the next five years, Raman and his disciple Krishnan published this effect in 1928, in the Nature journal, describing a new type of secondary radiation [1].

In the bibliography, there are several works where another type of scientific instrumentation is used to develop different teaching units that serve to improve the understanding of chemical and physical processes previously worked in the lectures [2-4]. For this reason, the fact that there are no works related to Raman spectroscopy to work on concepts developed in experimental science class for Teacher training in Primary Education in formation, makes the proposal that is presented more important.

In this work, we present a didactic unit proposal, which will try to work on various concepts developed in the subject of experimental sciences for future teachers of Primary Education in formation.

Firstly, the first part, where the crystallization of various chemical compounds is carried out, will help to pinpoint possible doubts arising in the master classes of the chemistry part. Next, it is analyzed how to differentiate the crystals by means of their type of geometry in its crystallization based on geological concepts. Finally, the crystals are differentiated by another method based on the Raman spectroscopy previously described, where students can observe concepts of light treated in physics class such as lasers, and electromagnetic lengths, where for the case in which some crystals that are not perfectly crystallized, this portable equipment will be used. In this way, the students have their first experience of this type, and use real scientific instrumentation.

Methods

Teaching and Learning processes based on Constructivism theories and IKD model of the Basque Country University

The students can put into practice the solubility concepts in master classes, through activities based on the IKD didactic model of the University of the Basque Country and in constructivist models of teaching and learning [5]. Learning cycles have been developed and in a group, dynamic and cooperative way, the students explore their previous knowledge about solubility and crystallization, reflect on these ideas, make meaningful learning and apply these new learning in research contexts in the laboratory [6]. In particular it has been discussed in the classroom about the amount of salt (compound) that can be dissolved in water and has been investigated on the factors that influence the solubility and on the crystallization process.

Thanks to this first methodological phase, the students begin to experience the first fundamental concepts of solubility and crystallography and subsequently the fact of observing week after week the growth of the crystals themselves, can help the student to encourage possible scientific vocations, better understanding of theoretical master classes and letting them know how is working in the scientific field and spreading the importance of crystallography in our society

Instrumentation

A portable Raman spectrometer for the in-situ analysis in which samples are screened without being removed and a benchtop spectrometer for an in-depth study to identify the maximum number of compounds in each sample. The portable innoRam™ (B&WTEK$_{INC}$., Newark, USA) Raman spectrometer utilizes a 785 nm excitation laser and has a maximum output power of 300 mW. Variable attenuation of the output power was achieved through the implementation of filters, which allowed for the reduction of the output power down to 1% of the maximum. The spectra were typically collected using a wavelength range of 100-2200 cm^{-1}, although in some measurements larger ranges were used. The exposure time for each spectrum ranged from 2 to 10 s, and two to fifteen repeated acquisitions were averaged to improve the signal-to-noise ratio.

When we will work in-situ, measurements can be made directly on the surface of the crystals formed by the students using the probe that has the instrument, which transmits the laser emission throughout fiber optic In this case, the measurement area would be around 100 µm (see Figure 1), which it will be enough size for the type of crystals that the students have formed.

Figure 1: Portable Raman spectrometer with its probe for in-situ analyses

Methodology and experimentation

The development of this proposal will be carried out in three clearly differentiated phases: Salts crystallization based on Chemical theories, crystal identification based on Geological theories and crystal identification based on Physical-Chemical theories.

Salts Crystallization processes based on Chemical theories

In the first phase, students should prepare three different types of mineral crystals. For carried out this objective, they will prepare three solutions in water until reaching saturation. In this way, students will begin to use the laboratory material studied in the theoretical classes, using and observing its characteristics, and in the same way trying to correlate the concepts of chemical saturation previously learnt in master classes.

The solutions previously selected by the teaching staff, will be prepared, using a sodium nitrate ($NaNO_3$), potassium nitrate (KNO_3) and ammonium hydrogen phosphate ($NH_4H_2PO_4$) reactives. For the solutions preparation, in all three cases they will be carried out in the same way: In a beaker, 1 liter of Milli Q water will be heated previously. Later, this hot water will be poured on small containers where the students will try to dissolve the three compounds until reaching saturation. Once the solutions are dissolved, it will be reheated to approximately 70-80 °C, reaching again the saturation point. In most cases, the students will dissolve approximately 300 g of each compound. In this way, achieving in this way high quality crystallizations. To achieve optimum crystal nucleation, teaching will add to each solution a small mineral crystal and over time, the temperature will fall, leaving them at room temperature (about 20 °C) and will leave to stand in a safe place for their crystallization process during a period of approximately one month (see Figure 2).

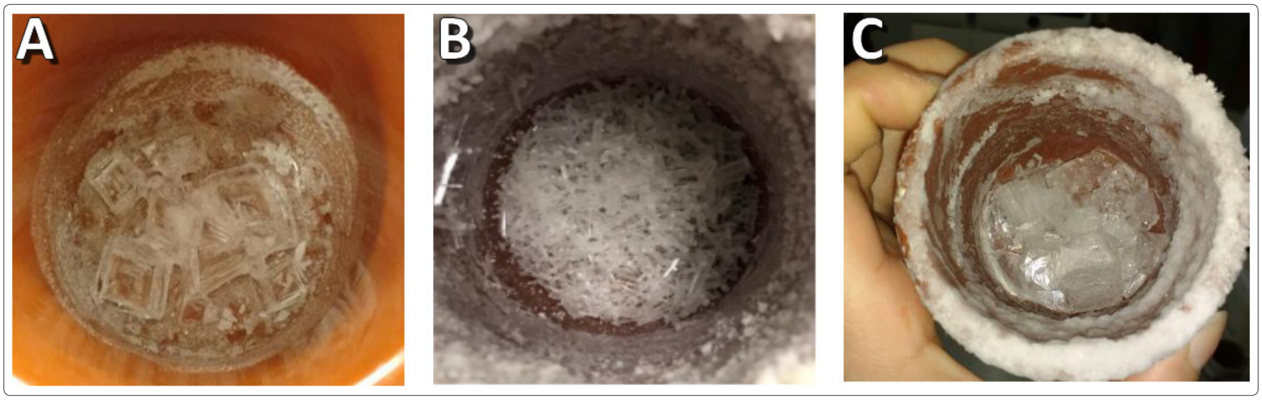

Figure 2: Crystals obtained by students: A) sodium nitrate ($NaNO_3$), B) potasium nitrate (KNO_3) and C) Ammonium dihydrogen phosphate ($NH_4H_2PO_4$)

In this way, the students can put into practice the solubility concepts seen in master classes, where water can only dissolve certain amount of salt (compound). Therefore, when hot water is used, it will dissolve more salt (compound) than cold water; so as the hot water cools, a part of the salt (compound) is released, and the other one that cannot dissolve, forms the solid crystals. Thanks to this first methodological phase, the students begin to experience the first fundamental concepts of crystallography and subsequently the fact of observing week after week the growth of the crystals themselves, can help the student to encourage possible scientific vocations, better understanding of theoretical master classes and letting them know how is working in the scientific field and spreading the importance of crystallography in our society.

Crystal identification based on Geological theories

In a second phase, once that month passed, the students will observe the crystals that have grown inside their containers. Firstly, one of the objectives will be to observe what kind of crystals have formed. For the observation methodology and subsequent for the description of them, teacher will give some guidelines to distinguish the formed crystals according to their geometry based on the geological morphology.

In this way, the students can observe how the $NaNO_3$ crystals form a trigonal system where their angles will be ≠ 90º and all the crystal sides are equal, that is, a = b = c. Moreover, for the case of KNO_3, students will observe their orthorombic system, where all their angles are right (=90º) and the crystal sides are all different (a ≠ b ≠ c). Finally for the case of $NH_4H_2PO_4$, students will observe how they form crystals in a tetragonal system, where two of their sides are equal and one is different (a = b ≠ c) and additionally, their three angles are 90º (see Figure 3).

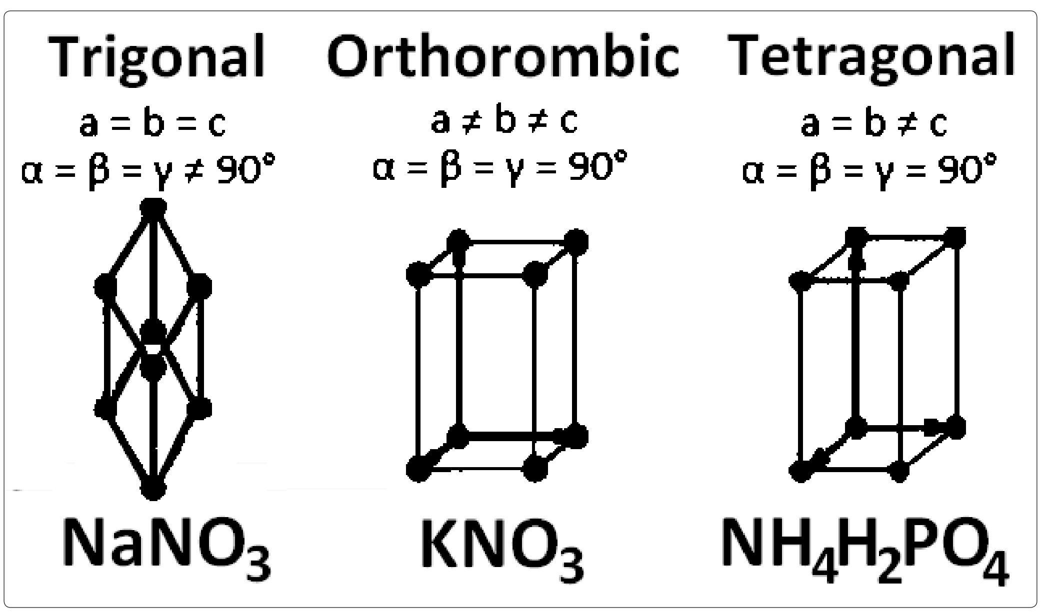

Figure 3: Geological geometry for the three used compounds observing their Trigonal, Orthorombic and Tetragonal systems.

Thanks to this second phase in the proposed methodology, the fact of developing the crystallography thematic can promote skills such as visual and spatial perception. Surprisingly, crystallography and subsequently the crystallization technique is a topic that is often absent in the majority of educational programs in Primary Education, so the development of these types of topics gets high importance for the educational development of the students.

Crystal identification based on Physical-Chemical theories

Finally, in a third phase, in some cases in which, after one month, the crystals have not been developed as desired, we will use the portable Raman spectroscopy instrument previously described to illustrate students, the possibility that exists to be able to identify in a scientific way the crystals that have tried to form and they have not achieved it.

To carry out this, teachers will gather the crystals formed not properly deleting the name of the compounds, so there will be three test samples that students will not know what kind of crystals correspond to each compound. In class, the students will meet in groups so that each and individually (group) has the possibility of differentiating the crystals using Raman spectroscopy. The fact that this technique provides a specific vibrational information of the molecules´ bounds, in a certain way, this technique also gives us the possibility of having a "fingerprint" of the compound in study in a simple way (see Figure 4). This type of technique consists in that the laser of the equipment (in our case red laser) when striking on the crystals promotes an excitation of the molecules of the own crystal, that can vibrate, rotate etc. This type of excitation is translated into a spectrum (different peaks) that is displayed on the screen of a computer connected to the Raman spectrometer. These peaks refer to different vibrational modes of the molecules, so that each of the bands of each spectrum, corresponds to different parts of the molecule, so as it has been mentioned above, each of the crystals has its own fingerprint.

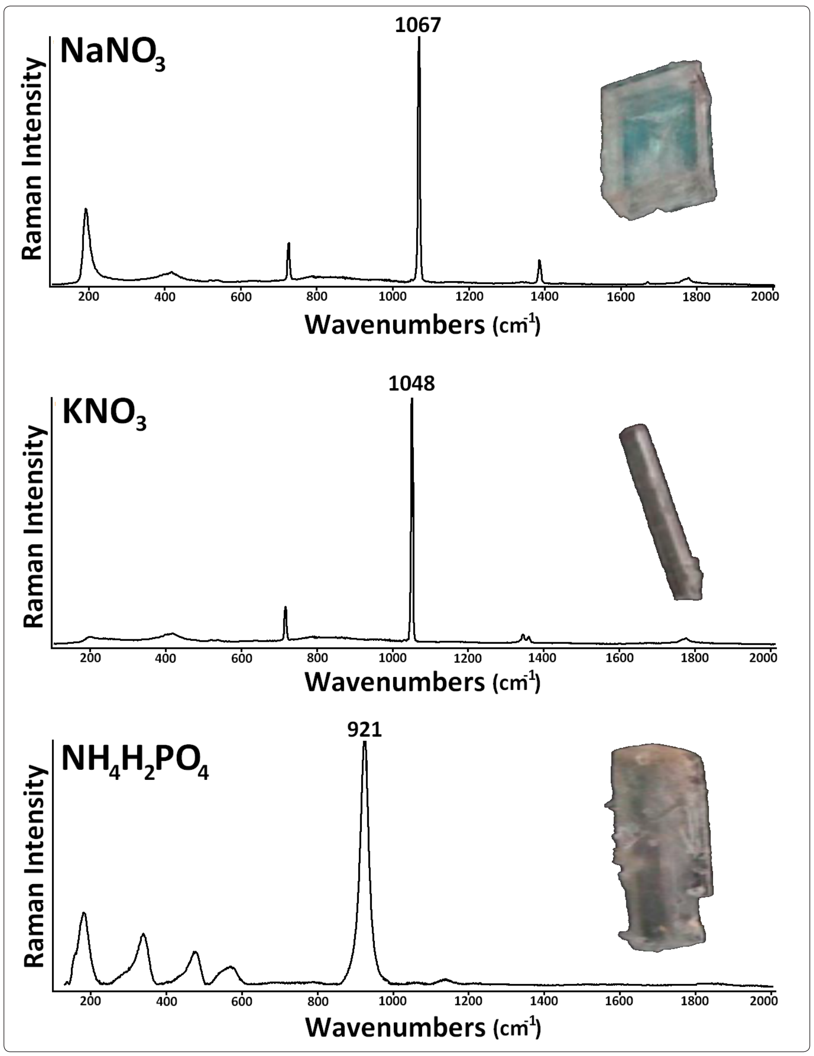

Figure 4: Raman spectra for the three types of crystals, showing their main raman peaks, which helps to students for their simple identification (for the case of $NaNO_3$, main raman band at 1067 cm^{-1}; for KNO_3, main raman band at 1048 cm^{-1} and for $NH_4H_2PO_4$, main raman bands at 921 cm^{-1} respectively).

Thus, as can be seen in Figure 4, each of the crystals compounds, have a characteristic Raman spectra, which give them the necessary information to distinguish the different crystals. In this way, depending on the wavelength of the main bands of the compounds (high intensity peak) (see Figure 3), the students will differentiate the type of compound that they are analyzing, being able to differentiate them quickly and simply. Therefore, $NaNO_3$ presents its main Raman band at 1067 cm^{-1}, KNO_3 at 1048 cm^{-1} and $NH_4H_2PO_4$ at 921 cm^{-1}, respectively (see Figure 4). Thanks to this type of techniques, students can have a dynamic and attractive vision of science, technology and innovation.

Finally, thanks to the visualization of these spectra, students can relate the type of movements that occur in the materials particles, in this case crystals, where the concept of particles movement, which is quite abstract, can be understood. In addition, the fact of lasers use in a realistic way helps also students to understand how these kinds of concepts exist in the reality and are not science fiction. Finally, in addition to the aforementioned, the use of this type of instrumentation in television series such as CSI, for example, means that students pay more attention in classrooms, and help them to grow a basic scientific curiosity in their professional work, that is, in the Primary Education classrooms.

Conclusions

The methodology proposal presented in this work, based on IKD model explained above, will offer to Primary Education degree students a great possibility of applicability as a teaching resource, in which the fact of using Raman spectroscopy as a real scientific instrumentation can fill them with curiosity, amazement and interest. Moreover, this technique cannot only be used as a complement to this type of work, but also for didactic innovation projects and research projects. Thus, the fact of being able to use this type of tools means that the students are stimulated by their curiosity and desire to advance and learn, progressing in their scientific concern and therefore, improving the delivery of their future classes in a more motivated, didactic and rigorous way.

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