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**ORIGINAL ARTICLE**

# Enhancing pharmaceutical analysis laboratory classes via the measurement of minerals in Berocca® effervescent tablets

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**Abstract**
The improvement of teaching to support students' self-directed learning skills in pharmacy should include a professional approach. This relates the academic theoretical knowledge to the challenges in real working life. In pharmacy, these viewpoints are easily allied. Examples of real pharmacy practices give students opportunities to understand the significance and meaning of the theoretical aspects. This article describes the analysis and quantitation of minerals (sodium, calcium, magnesium and zinc) present in the Berocca® effervescent tablets using the atomic absorption (AAS) and atomic emission spectrophotometric (AES) methods. The concentrations of the minerals can be examined feasibly in the pharmacy undergraduate laboratories as the experiment is straightforward, logistically unchallenging, inexpensive and utilizes a pharmaceutical product that is readily available at the retail pharmacy. As the scope of the practical experiment is applicable to the real life pharmaceutical quality control process, students gain the academic, psychomotor and affective knowledge domains of the subject matter.

**Keywords:** *Atomic absorption spectrophotometry, atomic emission spectrophotometry, minerals, pharmaceutical analysis*

## Introduction

Pharmacy education should correspond to the needs of working life and foster a good quality of learning in order to produce pharmacy experts for challenging and multidisciplinary assignments. In addition to the content of the curriculum, it is necessary to pay attention to pharmacy teaching itself. Recent studies clearly show that teaching methods and teaching ideology are related to learning outcomes (Kember & Kwan, 2000; Prosser, Ramsden, Trigwell, & Martin, 2003). In the "hard" sciences, including pharmacy, teachers have a greater propensity to adopt a teacher-centered approach to teaching (Prosser et al., 2003; Lindblom-Ylänne, Trigwell, Nevgi, & Ashwin, 2006). The result is similar to the study of Finnish pharmacy education where the students are encouraged to memorize facts (Nieminen, Lindblom-Ylänne, & Lonka, 2004). Teachers can help students learn more deeply, not by trying to change the students, but by changing their learning environment. This can be achieved by adopting a student-centered approach to teaching where the teacher pays attention to the students' active learning process that includes students' perceptions, activity, and understanding.

For the pharmaceutical analysis module conducted at the Department of Pharmacy at the National University of Singapore, the second year pharmacy undergraduates are expected to learn the analysis of pharmaceuticals via the use of functional group tests, ultraviolet (UV), infra-red (IR), atomic absorption (AAS) and atomic emission spectrophotometric (AES) techniques. The contents of these subjects are generally chemistry-based and technically-inclined. The goal of expertise and professional skills is supposed to be the source of students' motivation in studying pharmacy in an academic context. This

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is true especially for the pharmacy practice and clinical pharmacy modules. For the pharmaceutical analysis and other non-clinical pharmaceutical sciences modules, the teacher however cannot assume that the motivation to achieve the learning objectives is linked with students' inner enthusiasm for the subject and not the teacher's action. Teachers should be reminded that students engage with the tasks if they increase the students' appetite for the subject. This will happen if students consider the learning task to be meaningful, important, and of value to them. For the non-clinical pharmacy modules, without a conscious connection of the theoretical concepts of the topics to real life applications, it is not uncommon to find pharmacy students questioning the purpose of learning the module.

## Background

For the Year Two undergraduate pharmaceutical analysis module, the course consists of lectures, tutorials and practical laboratory classes. For the practical laboratory classes, experiments related to each of the topics (functional group analysis, UV, IR, AAS and AES) were designed to be associated with real working life applications. In this paper, an experiment that was designed to enhance the AAS and AES practical laboratory classes is illustrated and discussed.

"Healthy" supplements containing natural products, vitamins and minerals are becoming increasingly popular. It is difficult to miss the ubiquitous advertisements in the media pertaining to the "wonder cures" of the health supplements that prevent damage to the body. It is widely accepted that minerals are important for the physiological functions of the human body. The major minerals (e.g. calcium, magnesium, phosphorus, sodium, chloride and potassium) are needed in the greatest quantities or are present in large amounts in the body. The three main functions of minerals are as constituents of the skeleton, as soluble salts which help control the composition of the body fluids, and as essential adjuncts to the action of many enzymes and other proteins. Zinc is a micromineral needed in the diet on a daily basis, but only in very small amounts (50 mg or less). Zinc is an important regulator of many genetic activities. Depression, lack of appetite, growth failure in children, and frequent colds and infections can also be symptomatic of insufficient dietary zinc. In terms of minerals, each Berroca® effervescent tablet (Bayer Healthcare, Cambridge, UK) contains: calcium—100 mg, magnesium—100 mg and zinc—10 mg. Sodium is present in the tablets however its amount is not cited on the product label. In the quality control (QC) of the Berocca® effervescent tablets, the levels of the minerals need to be routinely analyzed and quantified.

AAS and AES are used in the determination of metals in pharmaceuticals (Watson, 2005). In AAS, atoms of a metal are volatilized in a flame and their absorption of a narrow band of radiation produced by a hollow cathode lamp, coated with the particular metal being determined (e.g. magnesium or zinc), is measured. In AES, atoms are thermally excited in a flame (for example, flame photometry) so that they emit light and the radiation emitted is measured. While AAS measures magnesium and zinc, AES is suitable for the measurement of alkali metals such as sodium and calcium. As both AAS and AES techniques are adopted by the official monographs of the pharmacopoeias (for example, British Pharmacopoeia and the United States Pharmacopoeia) for the analysis of metals in pharmaceutical preparations, it is paramount for the pharmacy undergraduate students to learn and appreciate the concept and applicability of these techniques in pharmaceutical analysis.

In the practical laboratory classes, the most common and convenient procedure adopted for the analysis of metals using AAS and AES involves analyzing unknown samples or standard solutions spiked with the metals of interest. These test solutions can be prepared readily in the laboratories using metal standards. A possible concern of such experimental design is that students are often unaware of the origins of the samples and fail to appreciate the values of these spectrophotometric techniques in real life pharmaceutical analysis. This may lead to surface rather than deep learning of the subject. The new approach of analyzing the metals in the Berocca® effervescent tablets during the practical laboratory class is likely to substantially ameliorate this problem as the students are handling a real life pharmaceutical product. Furthermore, the Berocca® effervescent tablet contains metals suitable for both AAS (magnesium and zinc) and AES (sodium and calcium) and renders the experiment feasible to be conducted in the undergraduate laboratories.

## Laboratory experiments

### *Chemicals, glassware and equipments*

Berocca® effervescent tablets (Bayer Healthcare) were purchased at the retail pharmacy of the National University Hospital. Calcium, sodium, magnesium and zinc metal standards were supplied by BDH Chemicals Ltd (Poole, UK). All other reagents used were of analytical grade. 100 ml volumetric flasks, micropipettes (200 and 100 μl ranges) and beakers were provided to the students. Atomic emission measurement was performed using the PFP7 Flame Photometer supplied by Jenway Ltd (Essex, UK) while atomic absorption analysis was performed using the Perkin Elmer AAnalyst 100 system (Germany).

*Preparation of calibration standards and test samples*

The total number of students who participated in the practical laboratory classes was 103. The classes were conducted over 12 sessions and the time allocated for each practical laboratory class was three hours. Each session involved eight or nine students and they were further sub-divided into two working groups of four or five students per group. Prior to the start of the experiment, the students were given a short briefing. The students were provided with a scenario where they were the QC scientists performing analysis on a batch of Berocca® effervescent tablets. Each student group was provided with 1000 ppm (parts per million) or 1000 μg/ml stock solutions each of calcium and sodium and 100 ppm stock solutions each of magnesium and zinc. Using suitable dilution schemes, each group prepared three concentration levels of calibrants of each metal within the stipulated concentration ranges, namely, calcium (0–30 ppm), sodium (0–30 ppm), magnesium (0–1 ppm) and zinc (0–2 ppm). As the instruments possess relatively narrow dynamic range, the highest concentration prior to detector saturation for each metal had to be made known to the students. The test sample was a pre-dissolved solution of the Berocca® effervescent tablet (one tablet in 200 ml of water). Each group was reminded by the teacher to dilute the test sample solution suitably so that the expected instrumental measurement falls within the calibration curve. However, no detailed instruction was provided to the students with regards to the design and calculation of the dilution schemes for the calibrants and the test sample.

*Experiment (a): Measurement of calcium and sodium using AES*

The flame photometry instrument was set up in the presence, and with the involvement, of the students. A teaching assistant (graduate student) was present to supervise the experiment. However, the operation of the instrument was performed directly by the undergraduate students. The metals were analyzed sequentially by selecting the respective wavelength filters on the instrument for calcium and sodium. For the analysis of each metal, the students first measured the atomic emission values of the calibration standards and finally, the reading of the diluted test sample. The emission readings of the calibration standards were used to plot a calibration curve that was used subsequently for the calculation of the concentration of the diluted test sample. The final amounts of calcium and sodium present in each tablet were determined after taking into account the dilution factors.

*Experiment (b): Measurement of magnesium and zinc using AAS*

The atomic absorption spectrophotometer was set up with the direct involvement of the students. Similar to AES, the operation of AAS was conducted directly by the undergraduate students under the supervision of a teacher. The metals were analyzed sequentially by selecting the respective hollow cathode lamps for the analysis of magnesium and zinc. The analytical procedures of AAS were similar to that of AES, except that the absorption readings of the calibration standards were used to plot a calibration curve automatically and the concentration of the diluted test sample was measured and reported directly by the instrument. The final amounts of magnesium and zinc present in each tablet were determined after taking into account the dilution factors.

## Results and discussion

*Feasibility of Berocca® tablet experiment in undergraduate laboratories*

The Berocca® effervescent tablet adopted in the experiment was found to be highly suitable for the undergraduate practical laboratory classes. The product is relatively inexpensive and is readily available at the retail pharmacy as an over-the-counter product. This implies that the procurement of the tablets by the laboratory officer for the practical classes is straightforward. The tablet contains four minerals that can be analyzed by both AAS and AES. This circumvents the handling of more than one pharmaceutical product for the experiment that may render the practical classes challenging from a logistic perspective. While the product is presented as a solid dosage form, the effervescent tablet is readily soluble in water and can be used directly for the experiment without further sample pre-treatment. This is important as each practical class is relatively short (3 h) and any elaborate sample pre-treatment steps would render the experiment tedious and ineffective. Being present as a solid dosage form, the product is relatively stable compared to liquid formulations and can be used for many sessions of the practical classes. Most importantly, the pharmaceutical product, unlike spiked samples, provides a meaningful linkage between the theoretical concepts and applications of AAS and AES.

*Motivation and learning of knowledge*

Academic students do not automatically have a higher level of thinking skills which are requirements for the successful studying at the university. In order to enhance the development of such skills, the teacher's responsibility is to provide the support that creates stimulating learning environments and contexts. In the practical laboratory classes, the students were presented with a clear picture of the potential applications of the AAS and AES techniques that they were learning. The teacher's practices concentrated on activating methods, such as discussion and group tasks, so that the interaction between teacher and students was emphasized. The outcome of this approach was positive as the students were clearly motivated and engaged with the teacher and demonstrator actively during the experiments. As the students were given minimum instruction on the dilution schemes, each group of students had to discuss and strategize the dilution of the calibration standards and samples among themselves prior to the start of the experiments. This teaching approach proved to be an encouragement of the students towards self-directed learning. In this case, the brief moments of pondering and discussion over the dilution issue in the small groups, did not require much extra work but brought considerable added value to the teaching. First, the students learned and appreciated the importance of working as a team player, a valuable asset to be acquired and applied in their future careers. Second, the students were motivated when the dilution schemes derived by them were given a positive acknowledgement by the teacher. Third, this approach increased the students' responsibility for their active role in quality learning.

The theoretical concepts of AAS and AES in pharmaceutical analysis are relatively remote and technical for the pharmacy undergraduate students. During the practical laboratory classes, students were tasked to operate the instruments themselves under the supervision of the teacher. This teaching approach was important as it enhanced the students' psychomotor domain of the knowledge in the subject and as a result, reinforced the academic domain of the knowledge learned during the lectures. Figure 1 illustrates the final class results of the measurements of the sodium, calcium, zinc and magnesium present in the Berocca® effervescent tablets. Such analytical reports simulate the QC results obtained in the pharmaceutical working environment. As the nature of the experiments served to relate the academic knowledge to the real working life application, the practical classes elevated the students' affective knowledge domain of the subject and allowed the students to develop a passion in pharmaceutical analysis.

As the work presented in the paper was conducted prospectively, it was challenging to compare the learning outcome of the new pedagogical approach with the traditionally adopted exercise quantitatively. However, under the university's guidelines, the students were required to provide feedback related to their learning outcome at the end of the semester. Six questions were posed for the students to assess and submit their ratings online (see Figure 2).

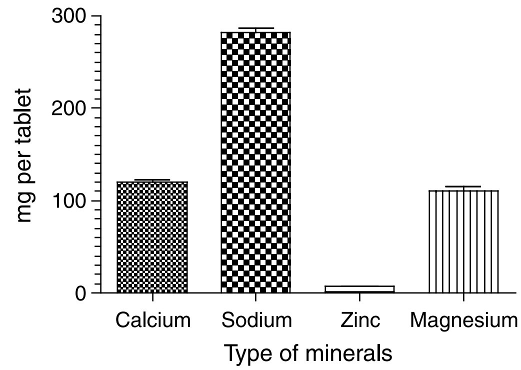


Figure 1. Plot of concentration (mg/tablet) against types of minerals present in the Berocca® effervescent tablets ($n = 103$).

A 5-point scale is used for the scores. The higher the score, the better the rating. The results of the feedback are presented in Figure 2. The faculty members' average score is the mean of all the scores for each question for the faculty member. The department average score is the mean score at the same module level (level 2000) within the department while the faculty average score is the mean score at the same module level (level 2000) within the faculty.

While the students' ratings were provided with respect to the entire course, including lecture, practical class and tutorial, it was assumed that the students' learning experience derived from the Berocca® effervescent tablet analysis experiment influenced their ratings on the faculty member. Among the six questions, Questions 4 and 6 were relevant to the learning outcome with respect to the new pedagogical approach. From the results obtained on Question 4, it was clear that the module increased the students' interest in the subject significantly as the

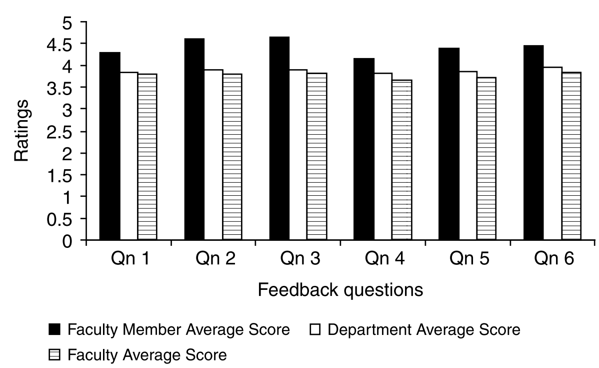


Figure 2. Students perceptions of the quality of faculty members' teaching during the semester. The six questions posed to the students: (1) The teacher has enhanced my thinking ability. (2) The teacher provides timely and useful feedback. (3) The teacher is approachable for consultation. (4) The teacher has increased my interest in the subject. (5) The teacher is able to demonstrate cross-disciplinary relationships in relevant topics and has taught us to draw interconnections between different areas in science. (6) The teacher is able to illustrate some actual or potential applications of knowledge covered in the syllabus.

score for the faculty member surpassed the ratings at both the department and faculty levels. This finding was consistent with the aim of the Berocca® effervescent tablet analysis experiment, which was to motivate the students in learning the pharmaceutical analysis subject. Increasing the interest of the students in the subject was important to promote self-directed learning. As the score for the faculty member based on Question 6 was also higher than the average scores measured at the department and faculty levels, the results indicated that the students appreciated the illustration of actual or potential applications of knowledge covered in the syllabus. One key example of such a pedagogical approach was the Berocca® effervescent tablet analysis experiment where the potential application of AAS and AES methods in pharmaceutical analysis was exemplified.

## Conclusion

The Berocca® effervescent tablet analysis experiment was found to be suitable and feasible for the pharmacy undergraduate AAS and AES practical laboratory classes. The teaching approach connected the students' academic knowledge to real working life application and motivated the students towards student-centered learning. As students see and understand the association between practice and theory, they learn to respect the significance of theoretical pharmacy studies. The responsibility of students' motivation is on the teachers—good teaching awakens motivation.

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