Misconceptions and metaconceptions in instrumental analysis

Antonio Doménech-Carbó
José Vicente Gimeno-Adelantado
Francisco Bosch-Reig

ABSTRACT

Normative science models are accompanied by a number of metaconceptions dealing with the foundations, methods and applications of the fundamental science formulations whose presence in the science curricula is needed. In the case of instrumental analytical chemistry, metaconceptions include from analytical properties to operational concepts. Student's misconceptions concerning instrumental methods, procedures and protocols have been established from coordinated tests and tutorial interviews during laboratory lessons. Results for chemical engineering university students indicate that most misconceptions in instrumental analysis can be associated to a non-structured view of concepts and methods in turn related with metaconceptions in analytical chemistry.

Keywords: Misconceptions. Metaconceptions. Instrumental analysis. University.

Concepções errôneas e metaconcepções em análise instrumental

RESUMO

Modelos normativos de ciência vêm acompanhados de um número de metaconcepções que tratam das fundamentos, métodos e aplicações fundamentais de ciência cuja presença nos currículos científicos é necessária. No caso de química analítica instrumental, metaconcepções incluem desde propriedades analíticas até conceitos operacionais. Erros conceituais dos estudantes no se refere a métodos instrumentais, procedimentos e protocolos foram estabelecidos através de testes coordenados e entrevistas dirigidas durante aulas de laboratório. Resultados para estudantes de engenharia química indicam que a maioria dos erros conceituais em análise instrumental pode ser associada a uma visão não estruturada de conceitos e métodos que por sua vez estão relacionados com metaconcepções em química analítica.

Palavras-chave: Concepções errôneas. Metaconcepções. Análise instrumental. Universidade.

INTRODUCTION

Analytical chemistry can be considered as one of the essential branches of chemistry. As far as analytical chemistry practice; i.,e., chemical analysis, is directly related with a

Antonio Doménech-Carbó – Departamento de Química Analítica, Universidade de Valência. Dr. Moliner, 50, 46100 – Burjassot (Valência), Espanha. E-mail: antonio.domenech@uv.es
José Vicente Gimeno-Adelantado – Departamento de Química Analítica, Universidade de Valência. Dr. Moliner, 50, 46100 – Burjassot (Valência), Espanha. E-mail: jvgimeno.adelantado@uv.es
Francisco Bosch-Reig – Departamento de Química Analítica. Faculdade de Química, Universidade de Valência. Dr. Moliner, 50, E-46100 – Burjassot (Valência), Espanha. E-mail: Francisco.bosch@uv.es

wide variety of social demands (from environmental analysis to biomedical analysis, quality assessment in industry, etc.), learning analytical chemistry and chemical analysis involves not only a series of 'chemical' concepts, but also a series of metaconceptions dealing with the scope, strategies, methods, etc. that are specific of analytical chemistry.

The importance of analytical chemistry in the context of teaching chemistry has been largely recognized in recent educational research (MURRAY, 1989). Roughly, one can discern between 'classical' or 'non-instrumental' analytical chemistry and 'modern' or 'instrumental' analytical chemistry. The former is usually focused in the study of ionic equilibria in aqueous solution, volumetries and gravimetries, while the second incorporates optical, magneto-optical, electrochemical methods conjointly with separation (chromatography, electrophoresis) and mass spectrometry methods (BRAUN, 1986; LAITINEN, 1989; SOMMER, 1993; KELLNER, 1996). Several educational approaches have been proposed for teaching analytical chemistry, namely, complementary use of laboratory-based (FITCH et al., 1996) and coupled theory/experimentation (WRIGHT, 1996) methodologies, as well as so-called investigative (KLOCKOW, 1981; WENZEL, 1995) and technological (SMITH; STOVALL, 1996) approaches.

Analytical chemistry learning can be viewed within the general frame provided by recent research in science education. In particular, along the last decades, a considerable research in science education has been devoted to characterize misconceptions, spontaneous conceptions or alternative conceptions influencing learning (DRIVER, 1981; GILBERT; WATTS, 1983). Here, the term misconceptions will be used for designing mental constructs dealing with concepts, terms, relations, procedures, etc. involved in science which: i) can be considered as erroneous to any extent, and, ii) result from spontaneous reasoning of the students.

In this context, it should be noted that analytical chemistry involves, apart from having concepts, conventions, terminology, laws, procedures, etc. In common with other branches of chemistry, a definite set of highly specific concepts, methods, etc. (DUSCHL, 1985; HODSON, 1985; GILBERT, 1992). Most of these concepts and procedures deal with the so-called analytical properties (accuracy and precision, reproducibility, etc.), operational concepts (separation, identification), mathematical tools (linear correlation, integration, derivation) which have to be integrated with social demands for structuring chemical analysis. Much of these items are characterized by: i) to be tailored with other related concepts and procedures; ii) involves 'something more' than strict chemical concepts for which formal definitions hold. In order to separate it from specific science concepts, it will be termed here as metaconceptions.

In this sense, approaches for teaching of analytical chemistry should consider misconceptions dealing with not only specific concepts and methods, but also with metaconceptions, in order to provide a more complete view of this branch of chemistry. For instance, the concept of accuracy (defined as the degree of similarity between the obtained result and the 'true' result in an experiment) in chemical analysis is lied with the use of standards, but also with a demand of analytical methods that implies the adoption of determined analytical strategies, for instance, the inclusion of calibration steps with appropriate standards in the analytical protocols. Students misconceptions in chemical equilibrium (VOSKA AND HEIKKINEN, 2000; WHEELER AND KATZ, 2006), errors (TOMLINSON et al., 2001) and near-infrared spectroscopy (DIFOGGO, 1995) have been recently reported.

Accordingly, learning analytical chemistry involves not only the knowledge and use of the concept of accuracy, but also of its more wide meaning as metaconception in this scientific frame. This means that one can expect that students misconceptions can affect not only orthodox science concepts, relations, procedures, etc., but also metaconceptions. Students misconceptions on instrumental analysis can be viewed, at least partly, within the frame provided by Taber (2002) and Mabrouk (2002) on chemistry misconceptions and Cousin (2006) and Meyer and Land (2003, 2006) on treating threshold concepts. This situation is comparable with that previously described for the definition of fundamental science concepts such as mass; here, ontological aspects play an essential role in students understanding of such concepts (DOMÉNECH, 1992, 1997; DOMÉNECH et al., 1993). As recently reported by Davidowitz et al. (2001), the reasons given by second year chemical engineering and science students for making repeat measurements, the majority of students perceive the purpose to be either to identify a recurring (correct) value or to perfect measuring skills.

The current report is devoted to discuss the meaning of such metaconceptions in a sub-field of analytical chemistry: the so-called instrumental analysis, and to obtain information on misconceptions dealing with such metaconceptions. For this purpose a field study was performed with a sample of 25 students of Chemical Engineering (3rd year) from the University of Valencia, based on structured interviews during laboratory sessions (Instrumental Analysis lessons) along the ordinary 2007-2008 period.

MISCONCEPTIONS ON INSTRUMENTAL ANALYSIS

Analytical chemistry involves, apart from formal, involving specific concepts and formulations within the general frame of chemistry a series of peculiar metaconceptions which can be associated to aims and capabilities of analytical chemistry and chemical analysis and its social frame (DOMÉNECH et al., 2008). These metaconceptions are summarized in Chart 1.

Chart 1. Summary of metaconceptions in analytical chemistry and chemical analysis.

Metaconceptions on	Concrete aspects
Aims and capabilities of analytical chemistry and chemical analysis	- The generalized view of chemical species and the general concepts on identification, characterization, speciation and determination. - The possibility of using any physico-chemical signal as a source for analytical information. - The possibility of analyzing macro-, micro-, and submicro-systems and short-time intermediates. - The generalized concepts of surface and structure analysis. - The possibility of analyzing complex multicomponent systems. - The possibility for analysis in situ and at a distance (teleanalysis).

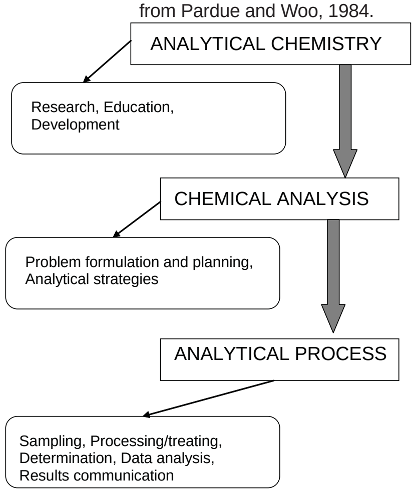
Metaconceptions on	Concrete aspects
Social frame where these fields are implemented	- Contributions to research in analytical chemistry itself other chemistry fields. - Normalized chemical analysis in biochemistry, pharmacology, toxicology, forensic sciences, etc. - Normalized chemical analysis for industry. - Analytical strategies and operations for environmental chemistry. - Analytical methods for archaeometry, conservation and restoration of pieces and materials forming the cultural heritage.
Formal and operational aspects involved in analytical chemistry and chemical analysis	- Metaconceptions dealing with general concepts: chemical analysis, operations, analytical process, analytical strategy. - Metaconceptions dealing with specific concepts: sampling, separation, identification, quantitation, etc. - Metaconceptions dealing with specific methods, and operations: titration, gravimetry, electrolysis, chromatography, etc. - Metaconceptions dealing with the so-called analytical properties: accuracy, precision, reproducibility, etc. - Metaconceptions dealing with social/analytical demands: economy, robustness, safety, etc.

Recent developments/tendencies in analytical chemistry involving chemometric methods, quality assessment, computerization, advanced sensing and transduction, miniaturization, hyphenated analytical techniques, robotization and automation can also be included in the domain of metaconceptions in analytical chemistry.

Several aspects can be remarked with regard such metaconceptions:

a) Several concepts in analytical chemistry should be hierarchically structured through several levels and functional processes (PARDUE; WOO, 1984; VALCÁRCEL, 1992; VALCÁRCEL; LUQUE DE CASTRO, 1995). A brief summary is depicted in Chart 2.

Chart 2. Hierarchical view of analytical chemistry, chemical analysis and several important concepts. Adapted from Pardue and Woo, 1984.



b) Metaconceptions involve not only concepts sustained by concrete definitions (accuracy, limit of detection, linearity interval, limit of quantitation, standard deviation) but also concepts “in flux”, concerning operational and or relational definitions (trazeability, detection, determination).

c) Metaconceptions are in turn related with learning of operational skills. Thus, the selection of a given analytical strategy for solving a certain analytical problem is supported to a great extent by an efficient use of metaconceptions.

From an educational view, the relevant point to emphasize is that such conceptions can be considered as an essential addition to fundamental chemistry concepts. All these matters possess a special importance in the context of teaching instrumental analysis because this field occupies a prominent position in the analytical chemistry curriculum and focuses a significant part of research and practice in chemical analysis.

STUDENTS MISCONCEPTIONS ON INSTRUMENTAL ANALYSIS

University students misconceptions regarding instrumental analysis were studied from a sample of 25 Chemical Engineering students (3rd year) from the University of Valencia, using an interview protocol during laboratory sessions (Instrumental Analysis lessons) along the ordinary 2007-2008 period. The students were divided in two groups of 11 (group 1) and 14 (group 2) students, respectively. The structured interviews comprised three questionnaires Q1, Q2 and Q3, to be completed in the analytical chemistry laboratory during regular classes. Figures included in such questionnaires are depicted as Figs. 1, 2 and 3.

Questionnaire Q1

Q11.- Indicate three analytical methods involving ‘advanced’ instrumentation.

Q12.- We dispose of a pH-meter for determining the acidity of an aqueous solution. Describe (briefly) the sequence of <u>all</u> operations to be performed.

Q13.- Indicate the result obtained in your measurements.

Questionnaire Q2

Q21.- Provide a description of phenomena involved in spectrophotometric measurements.

Q22.- Describe all operations involved in the record of the absorption spectrum in the visible region of a colorant X in solution.

Q23.- The figure shows the absorption spectrum obtained in an aqueous solution of a colorant X (squares) and that of an unknown substance Y (rhombs). Describe the characteristics of this spectrum of X and indicate the color of the colorant.

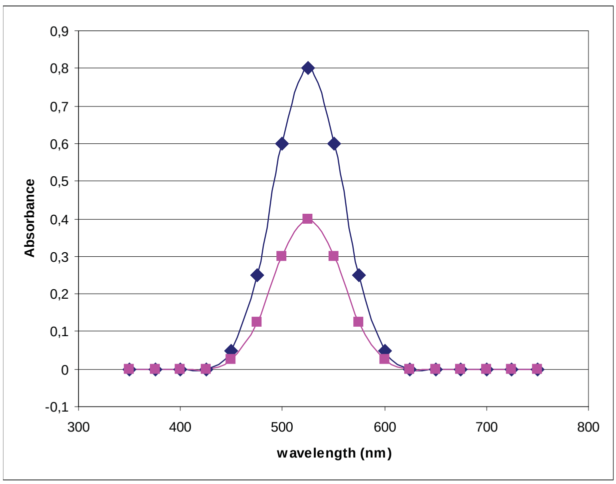
Q24.- Indicate if the second spectrum (Y) can correspond to a diluted solution of the colorant X. (Two options, A and B).

Questionnaire Q3.

Q31.- Draw the spectra to be expected for more and more diluted solutions of the colorant X.
Q32.- Obtain the spectra of a set of solutions of Ponceau S in concentrations 0.1, 0.2, 0.4, 0.6 and 0.8 mM were used). Indicate a method for establishing the concentration of a problem solution using the above data.
Q33.- The figure (Figure 2) shows the absorption spectra obtained in aqueous solutions of two colorants X (squares) and Y (rhombs). Describe the characteristics of these spectra and calculate the pertinent parameters characterizing such spectra.
Q34.- Indicate if the second spectrum (Figure 3) can correspond to a mixture of the colorants X and Y.

Figure 1. Spectra accompanying Questionnaire Q2.

Option A:



Option B:

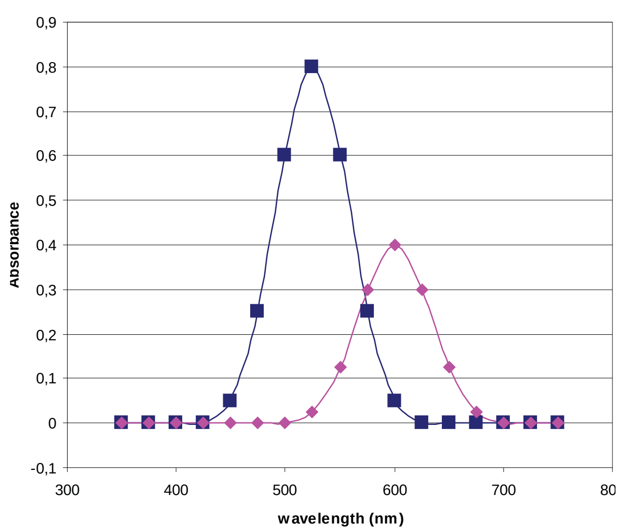


Figure 2. Spectra accompanying item Q33.

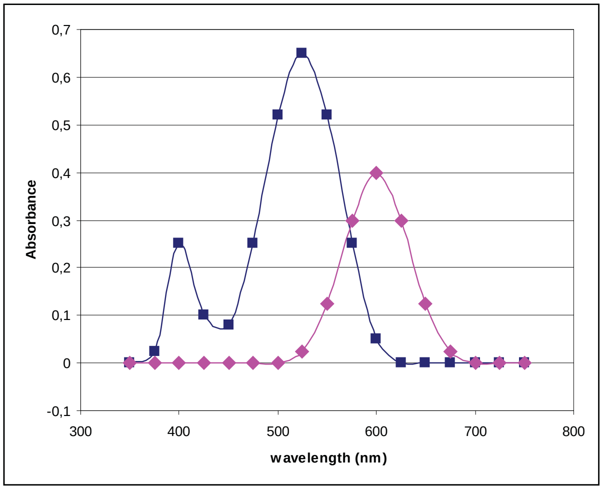
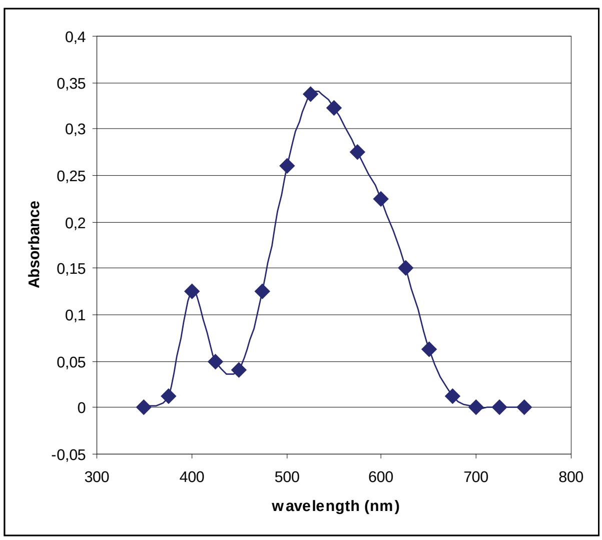


Figure 3. Spectra corresponding to item Q34.



A mixed written response/interview protocol was used. Each one of the items of the Questionnaires was first accomplished by the students and immediately their responses were commented and discussed in group. Individual responses were recorded conjointly with observations and comments along the session. The structure the research protocol is described in Charts 3, 4 and 5 for Questionnaires Q1, Q2 and Q3, respectively. The students were told that this study was part of educational research that was intended to help improve their instruction and that its outcome would have no effect on their grades. Ordinary pH-meters and single-beam educational spectrophotometers were used by the students during the sessions.

Chart 3. Structured interview/questionnaire Q1.

Question	Student's action	Interviewer participation
Q11. Citation of known instrumental methods	Written response	Data comment, citation of a more extensive list of instrumental methods Distinction between methods, procedures and protocols
Q12. We dispose of a pH-meter for determining the acidity of an aqueous solution. Describe (briefly) the sequence of all operations to be performed	Written response	Asking about the validity (accuracy and precision) of the results
	pH measurement	Discussing with the students the need for calibration (for accuracy) and repeating measurements (precision). Finally, claims the expression of the final result of the measurement series (correct expression of the result).
Q13. Indicate the result obtained in your measurements	Calculations	Comment on responses

Chart 4. Structured interview/questionnaire Q2.

Question	Student's action	Interviewer participation
Q21. Provide a description of phenomena involved in spectrophotometric measurements	Written response	Remembering and detailing concepts of spectrometry, colorimetry, spectrophotometry, absorption, reflection, diffusion, absorbance, etc.
Q22. Describe all operations involved in the record of the absorption spectrum in the visible region of a colorant X in solution	Written response	Discussing involved concepts (spectra, spectrophotometry/colorimetry, absorbance, Lambert-Berr law, etc.
	Record of the spectrum of a solution of a colorant	Discussing with the students the need for zero adjust, possibility of wavelength error (need of instrument calibration) and repeating measurements (precision).
Q23. The figure shows the absorption spectrum obtained in an aqueous solution of a colorant X (squares) and that of an unknown substance Y (rhombs). Describe the characteristics of this spectrum of X and indicate the color of the colorant	Calculations	Comment on responses Determination of characteristic parameters, wavelength at the maximum of absorbance (λ_{max}) and the coefficient of molar absorptivity (ε). Asking about the relation between the color color of the colorant and the spectrum
Q24. Indicate if the second spectrum (Y) can correspond to a diluted solution of the colorant X. (Two options, A and B)	Written response	Discussing responses and claiming for a 'methematical' method for ensuring the response

Chart 5. Structured interview/questionnaire Q3.

Question	Student's action	Interviewer participation
Q31. Draw the spectra expected for successively diluted solutions of the colorant X used in Q23	Written response / group interview	Verifying written responses and comments. Discussion on quantities to be used for satisfying Lambert-Beer law
Q32. Obtaining the spectra of problem solutions	Record of the spectra of Ponceau S solutions	Control of experiment and Discussing with the students the need for zero adjust, possibility of wavelength error (need of instrument calibration) and repeating measurements (precision).
	Group interview	Wavelength selection and analysis of absorbance/concentration data
Q33. Describe the characteristics of the spectra of X and Y colorants (Fig. 4)	Written responses / group interview	Comment on responses Determination of characteristic parameters, wavelength at the maximum of absorbance (λ_{max}) and the coefficient of molar absorptivity (ε). Asking about the relation between the color of the colorant and the spectrum
Q34. Indicate if the spectrum can correspond to a mixture of X and Y (Fig. 5)	Written response / group interview	Discussing responses and claiming for a 'methematical' method for ensuring the response Determination of the concentrations of X and Y

RESULTS

The obtained results for Questionnaires Q1, Q2 and Q3 are summarized in Tables 1, 2 and 3. No significant differences were observed between the results obtained for the groups 1 and 2 of students. First of all, it should be noted that the students developed during 2006-2007 a course on classical analytical chemistry where gravimetry and volumetry methods were extensively studied. Such methods are considered as 'classical' or 'non-instrumental'. Remarkably, the majority of the Q11 responses included 'classical', 'non-instrumental' methods like gravimetry or volumetry, so that, only ca. one half of the students indicated any 'genuine' instrumental method.

Table 1. Results on Questionnaire Q1. (*) Responses including non-correct terms such as "Electromagnetic" or "Calibration"

Item	Response	Number (percentage) Group 1 N = 11	Group 2 N = 14
Q11	Gravimetry	8 (72.7)	12 (85.7)
	Potentiometry	3 (27.3)	5 (35.7)
	Volumetry	6 (54.6)	7 (50.0)
	Spectrometry	7 (63.6)	8 (57.1)
	Chromatography	2 (18.2)	3 (21.2)
	Diffraction	1 (9.1)	0 (0.0)
	Non-correct (*)	2 (18.2)	4 (28.6)
Q12	Need for calibration	2 (18.2)	4 (28.6)
	Need for cleaning	3 (27.3)	4 (28.6)
	Need for repeating measurements	3 (27.3)	3 (21.4)
	Need for stirring	0 (0.0)	1 (7.1)
Q13	Mean value of 3-5 measurements	8 (72.7)	10 (71.4)
	Standard deviation	3 (27.3)	4 (28.6)
	Distinction between accuracy and precision	3 (27.3)	4 (28.6)

Table 2. Results on Questionnaire Q2.

Item	Response	Number (percentage) Group 1 N = 11	Group 2 N = 14
Q22	Need for zero correction	1 (9.1)	2 (14.3)
	Need for cleaning	3 (27.3)	5 (35.7)
	Need for repeating measurements	3 (27.3)	2 (14.3)
	Need for calibrating wavelength	0 (0.0)	0 (0.0)
Q23	Single peak aspect	2 (18.2)	5 (35.7)
	Absorbance maximum determined	3 (27.3)	5 (35.7)
	Correct color	0 (0.0)	0 (0.0)
Q24	Correct interpretation spectra in option A	11 (100)	14 (100)
	Correct interpretation spectra in option A	9 (81.8)	12 (85.7)
	Quantitative criterion	0 (0.0)	0 (0.0)

Table 3. Results on Questionnaire Q3.

Item	Response	Number (percentage) Group 1 N = 11	Group 2 N = 14
Q31	Essentially correct Other	9 (81.8) 2 (18.2)	12 (85.7) 2 (14.3)
Q32	Linear absorbance /conc. Plot Other	11 (100.0) 0 (0.0)	14 (100.0) 0 (0.0)
Q33	Double band for X and single band for Y Wavelength for all absorbance maxima determined Molar absorptivity at the wavelength for absorbance maxima determined	2 (18.2) 2 (18.2) 1 (9.1)	3 (21.4) 3 (21.4) 1 (7.1)
Q33	Correct interpretation of spectrum Quantitative criterion	7 (63.6) 0 (0.0)	7 (50.0) (100) 0 (0.0)

With regard to the operation with instruments, it should be remarked that the few students indicated the need for calibration, cleaning, repeating measurements and stirring during measurements in pH measurements. Similarly, few students indicate the need for previous zero adjust in spectrophotometric measurements and none consider the possibility of any instrument derive causing wavelength error. Interestingly, the percentage of responses claiming for the need for repetitive measurements in order to minimize non-systematic errors in Q22 are approximately half than those obtained in the case of pH measurements (Q12). This can be interpreted in terms of a certain idealized view of instrumentation: as more sophisticated instrumentation is, more precise should be it.

With regard to concepts involved in spectrophotometry (Q21), there was a set of vague responses regarding the general involved phenomena (distinction between light absorption, reflection and dispersion), its description in terms of modern physics (photon absorption, quantum transitions, etc.) and magnitudes and laws to be used (absorbance, Lambert-beer law, etc.).

Responses to items Q31 and Q32 were almost entirely correct. The students, however, confused in few cases the spectral plots with typical linear absorbance vs. concentration plots. Remarkably, the students do not express in precise way what absorbance has to be measured. Additionally, the students only consider the possibility of linear absorbance vs. concentration variations.

Qualitative description of spectra in Questionnaires 2 (Q23) and 3 (Q33) were surprisingly incorrect. The students do not remark the one-peak character of the represented spectrum for the colorant in Q23 (Fig. 1) and only in few cases determined the wavelength at the maximum absorbance as a part of their description. Similarly, in Q33, only a low percentage of students (ca. 20 %) make a description of the colorants in terms of two- and one-band features (Fig. 2).

In contrast, qualitative comparison of X and Y spectra (Q24) was almost entirely successful for both option A, where the spectrum of Y can effectively be described in terms of that for a more diluted solution of the colorant X, and option B, where different absorbing compounds are involved. However, the students were unable to propose a numerical method for properly establishing such statements.

The same situation appears in the responses to item Q34. Now, the percentage of students which describe qualitatively the spectrum in Fig. 3 as a combination of spectra in Fig. 2 ranged between 50.0 and 63.6 %, clearly lower than ca.100 % who correctly interpreted the spectrum in Fig. 1. As in the case of item Q24, the students do not propose a clear mathematical procedure for establishing the above statement.

DISCUSSION

University students exhibit a set of generic misconceptions on instrumental analysis. Firstly, formal misconceptions involved in instrumental analysis comprise:

- Concepts, terms and symbols involved in instrumental analysis are frequently used with low accuracy.

- The foundations of the instrumental method and the operation of the equipment are generally viewed as secondary or trivial insights during analysis.

Several specific misconceptions detected in instrumental analysis can be added:

- Equipment is frequently regarded as a exact, self-sufficient and semi-automatic device. Component degradation, biased measurements, etc. are in general not considered. Concomitantly, there is no need for cleaning, calibration, etc.

- There appears frequently a non-structured, non-integrated view of instrumental analysis within analytical methods.

- Students are aware of the importance of error consideration, but tend to identify manual errors in handling instrumentation as the main or even unique source of experimental error.

Such misconceptions can be ascribed to a cognitive style characterized by:

- A pseudo-empiricist view of science practice where apparently theory-independent measurements directly provide analytical information.

- A linear view of analytical strategies, so that analysis proceeds through a succession of operations (sampling, preliminary operations, signal measurement) with no need of revisions.

- A naïve view of instrumental analysis, based on an error-free assumption for data providing from instruments.

Current results, although preliminary, suggest that such misconceptions are directly related with the so-called metaconceptions in analytical chemistry and chemical analysis. Students appears to be adhered, to a large extent, to ritual (are able to perform superficial tasks and techniques to get a result, but fail to understand the complexity that lies behind it) and inert (concepts are understood but not actively used or connected to the 'real world') knowledge, a situation comparable with that described by Mabrouk (2002) and Taber (2002) on chemistry misconceptions and Cousin (2006) and Meyer and Land (2003, 2006) on treating threshold concepts. This means that, for instance, as far as chemical analysis is not viewed as a structured, complex process, operations involved in analytical processes are viewed as separated, self-consistent steps. From an educational point of view, it appears that attention should be paid to these subtle metaconceptions involved in analytical chemistry.

This approach can be considered as potentially interesting to examine students' misconceptions in other scientific branches, namely, Biology or Physics. As far as in general such disciplines also involve a complex lattice of related concepts and procedures accompanying formal definitions, laws, etc., it seems reasonable to expect that there is place for considering metaconceptions as a general issue influencing science learning.

CONCLUSIONS

Although instrumental analysis plays a significant role in chemistry and chemical engineering curricula, several student's misconceptions concerning instrumental methods, procedures and protocols can be detected. Using coordinated tests and tutorial interviews during laboratory lessons, a series of preliminary results on Chemical Engineering students reveals the appearance of severe misconceptions dealing with metaconceptions on analytical chemistry and chemical analysis.

Accordingly, learning analytical chemistry involves not only the knowledge and use of specific analytical concepts, but also of its more wide meaning as metaconception in this scientific frame. This means that one can expect that students misconceptions can affect not only orthodox science concepts, relations, procedures, etc., but also metaconceptions, conceptions associated to analytical properties, operational concepts and mathematical tools to be integrated with social demands for structuring chemical analysis and involving 'something more' than strict chemical concepts.

Student's misconceptions include a non-structured view of methods, procedures, etc., and erroneous view of concepts dealing with data treatment, errors calculation, relationships between variables. An empiricist, non-structured view of analytical chemistry can be detected where instruments are viewed as an autonomous, error-free 'black box' providing error-free data with no need of calibration or control, whereas weak relationships are established between data and aims along the analytical process. The current results suggest that educational approaches to instrumental analysis should incorporate an account for the metaconceptions involved in analytical chemistry and chemical analysis.

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