Quantitative microscale chemistry experimentation (volumetry, gravimetry, electrochemistry, thermochemistry)

Experimentos cuantitativos en química de microescala (volumetría, gravimetría, electroquímica, termoquímica)

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Abstract

Quantitative microscale experiments from China, Egypt, Germany and Macedonia dealing with electrochemistry, gravimetry and volumetry are described in this article. The experiments are low-cost, need no laboratory and can be performed as home experiments.

Key words: microscale chemistry, home experimentation, gravimetry, volumetry, electrochemistry, thermochemistry.

Resumen

En este artículo se describen los experimentos cuantitativos de química en microescala de autores de China, Egipto, Alemania y Macedonia sobre los temas de electroquímica, gravimetría, volumetría y termoquímica. Estos ensayos son de bajo costo, no requieren de ningún laboratorio y pueden ser realizados como experimentaciones domésticas.

Palabras clave: química en microescala, gravimetría, volumetría, electroquímica, termoquímica.

INTRODUCTION

Microscale Chemistry Experimentation (MCE) was introduced into research work by Pregl: In 1910 he microscaled Liebig´s quantitative analysis of the elements C and H using only 7 - 11 mg of an organic compound (GEKLE, 1994). In 1923 the Noble Prize for chemistry was awarded to him (http://www.nobel.se/chemistry/laureates/1923/press.html).

At school level MCE means working with substances in the volume range between 5 mL and 5 µL (1 micro drop). MCE saves chemicals, waste, space, time ("didactic money") and energy. Microscaling chemicals and materials increases safety, allowing experimentation even outdoors (WLOKA, 2002; SCHWARZ, 2002; SCHWARZ, 2003) thus supporting creativity and skills also of less self-confident girls and boys.

Student experiments are not popular in schools and colleges. This may be because they tend to be time-consuming and need expensive materials. Another reason: The groups must be small to be properly supervised. The importance of "learning by doing" as a first step of understanding is increasing. So more safe hands on experiments are needed which are low-cost and can even be performed at home.

Chemistry deals with matter - some thing that has a volume and a mass. So it is essential to introduce volumetry and gravimetry already during early childhood education (SCHWARZ, 2004$_1$; SCHWARZ, 2004$_2$).

MCE makes use of small disposable materials like injection bottles, blunted injection needles, infusion tubes, dropper bottles, dropper pipettes, 1- or 2 mL syringes, blisters and wellplates. These materials are standardized and mass produced resulting in cheap and useful equipment. For gravimetry a sensible Egyptian two-pan scale (SCHWARZ, 2004$_2$) and a digital multimeter with sensors for temperature and electricity are on the market (10 Eu each).

Very often the classes are too big for experiments done by pupils. Placing a wellplate 6 or even a transparent plastic surface on an overhead projector makes MCE suitable for demonstration even to the biggest auditory (EL-MARSAFY, 1995).

1. Quantitative electrochemical experiments with Wellplate
(Ning-Huai Zhou, Peter Schwarz)

Many kinds of wellplates with different sizes are available. COMBOPLATE® (http://www.microscalescience.com/home.htm) is spread in many countries. The authors of this and the following article prefer WELLPLATE 6 as it is cheaper and they think it is more versatile.

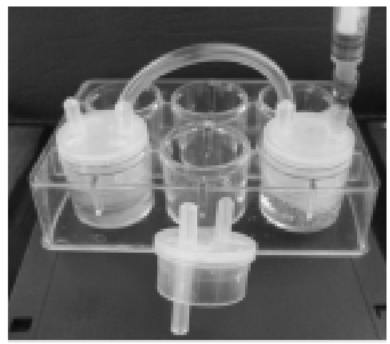


Figure 1
Wellplate 6 (WP6) with 3 of its 6 lids connected by short pieces of infusion tubing. Carbon dioxide produced from vinegar in a syringe and sodium carbonate in well 1 is tested by lime water in well 3.

1.1. Electric conductivities of Cola and Cola distillate (SCHWARZ 2004$_3$)

Electric conductivity is a property of solid or liquid substances which is measured under well defined conditions (AC current, defined size and distance of the electrodes…). In solids (metals) a flow of electrons closes the electric circuit, in liquids it takes place by anions and cations. The more ions a liquid contains the higher is its electric conductivity. Even in the best distilled water 10^{-7} mol/L of both ions are present. This measurable property of matter can be obtained by an ammeter allowing alternative current to flow between two injection needles dipping into wells of wellplate6 at constant distances.

The conductivity of Cola drink is mainly due to citric acid. Cola light contains phosphoric acid as an additional acid.

The quality of a microscale distillation of Cola can be tested: Cola distillate has about the same electric conductivity like demineralised water (0.03 mA AC at a voltage of 12 V).

Figure 2
Electric conductivity of Cola drink.

Figure 3
Electric conductivity of C. light drink.

1.2. Electrolysis of brine between pencil leads to obtain model fuel cells

- Cut off the tubes of 3 lids, use a glowing nail to weld 2 holes into each of them.
- Add 3 mL of brine [NaCl(aq)] to wells 1 and 2 and 3 mL of KI(aq) to well 3.
- Close the wells by the lids with the 6 electrodes.
- Assemble 2 *electrolytical cells* in series by connecting the electrodes in well 1 and in well 2 with a 9-Volt battery (Fig. 4).
- After 1 minute remove the 9-V-battery to stop the electrolysis.
- Test 1 of the self-made *battery of two model fuel cells* in series: Connect its electrodes 1 and 4 with a multimeter to measure the voltage (Fig 5).
- Test 2. Use this battery for electrolysis of potassium iodide in well 3 (Fig. 6).

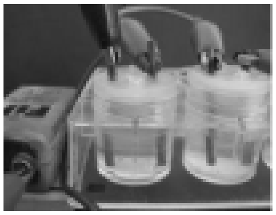


Figure 4
Electrolysis of brine between pencil leads (2 well in series).

Figure 5
Using wells 1 + 2 as Galvanic Cells red: $Cl_2(g) + 2e^- \rightarrow 2Cl^-(aq) + 2\,H_2O$
Ox: $H_2(g) + 2\,OH^-(aq) \rightarrow 2\,H_2O + 2e^-$

Results and explanation:

- The *model battery* made of *two Galvanic Cells* shows 4.86 V (Fig. 5). The redox potentials for the reactions $Cl_2 + 2e^- \leftrightarrows 2\,Cl^-$ and $H_2 + 2OH^- \leftrightarrows 2\,H_2O + 2e^-$ are 1.36 V – (-0.83 V) = 2.19 V. So the expected potential of two cells in series is 4.38 V.

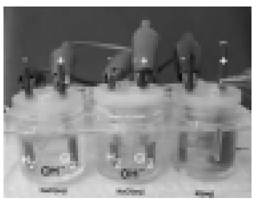


Figure 6
Electrolysis of potassium iodide in well 3 of wellplate6 taking wells 1 and 2 as power supply.

1.3 Coca Cola batteries

Coca Cola cans are made from two different metals depending on the country: in China aluminium is used, in Germany they are made from steel. Cola light is a good electrolyte. After being sandpapered such different Cola can metals connected to each other and surrounded by Cola drink as an electrolyte are a Galvanic Cell. These cells in series are batteries.

- Cut off the tubes from 3 lids, heat a screw driver and weld two parallel holes into each lid.
- Cut and sandpaper 3 strips of an aluminium can and 3 strips of a steel can. The strips must fit into the slits of the lids (Fig. 7).
- Assemble the materials like shown in Fig 8.

Figure 7
Materials for a Cola battery in a WP6.

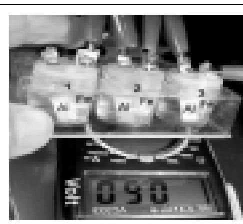


Figure 8
Potential in a 3-well Cola battery.

1.4 Half-cell reactions Zn/Zn^{2+} (anode) and Cu^{2+}/Cu in a single well

Standard reduction potentials are usually assigned in terms of the standard hydrogen electrode. For the following student experiments potentials are measured by combining half cells with different metals like zinc in 1M zinc sulfate solution and copper in 1M copper(II) sulfate solution.

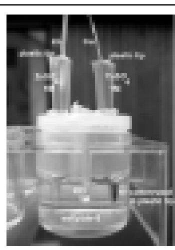


Figure 9
Zinc/copper half cells in one well of Wellplate 6.

Figure 10
Potential between Zinc/copper half cells in one well of Wellplate 6.

2. Measuring the change in enthalpy during the reaction of zinc powder and copper sulfate solution (W. Habelitz-Tkotz)

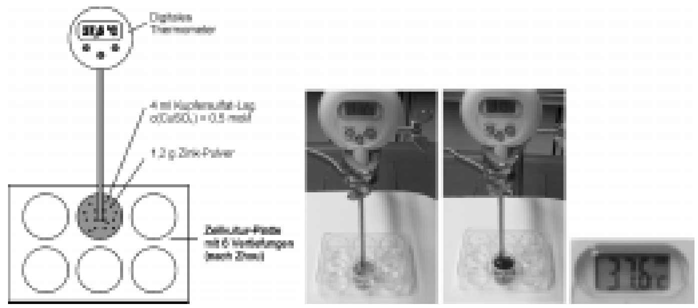
Materials:

Digital multimeter for temperature, Wellplate 6, stop watch, drinking straw spatula

Chemicals:

4 mL copper sulfate solution $c(CuSO_4)$ = 0,5 mol/L (1.248 g $CuSO_4 \cdot 5H_2O$ for 10 mL of solution), 1.2 g zinc powder.

Assembly:



Experiment:

- Transfer 4 mL of copper sulfate solution $c(CuSO_4)$ = 0,5 mol/L into well 2 of the wellplate.
- Dip the digital thermometer into the solution to measure the temperature J_0 of the solution.
- Add 1.2 g zinc powder and stir with the thermometer.
- Starting directly after adding the zinc powder read the temperatures any 15 seconds for about 5 minutes while you go on stirring. Write down the results.
  Present the results in two graphs.
  In the first graph: Plot the number of thiosulfate drops added versus time.
  In the second graph: $time^{-1}$ is plotted versus the number of thiosulfate drops added.
- Connect by a straight line.
- Find out the temperature directly after the end of the reaction by prolonging the line to the point of intersection with the y-axis

Special hints for carrying out the experiment:

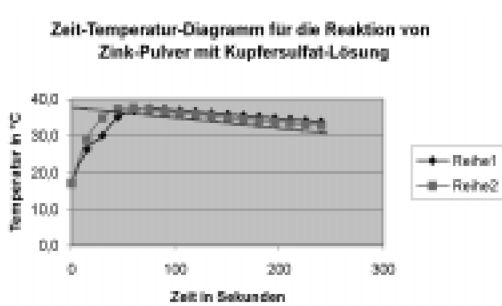
Do not replace copper sulfate by copper nitrate, because the nitrate ions cause side effects resulting in a strong increase of temperature.

You might repeat the experiment making a Daniell Cell and compare the electric energy released with the thermic energy. (Editor´s note: see 1.4 of the previous article).

Observations and results:

t in s	*E1* J *in* °C	*E2* J *in* °C
0	17,2	17,1
15	26,2	28,9
30	30,2	35,0
45	35,2	37,3
60	37,0	37,6
75	37,5	37,2
90	37,4	36,7
105	37,0	36,3
120	36,7	35,8
135	36,3	35,4
150	35,9	35,0
165	35,5	34,6
180	35,2	34,2
195	34,8	33,9
210	34,5	33,5
225	34,2	33,2
240	33,8	32,8

Change in temperature
$\Delta\vartheta = \vartheta_{max} - \vartheta_0 = 40\ °C - 17\ °C \approx 23\ °C$



Explanation:

Calculation of the quantity of heat Q_p released:

$Q_p = -[c_p(H_2O) \cdot m(H_2O) + c_p(Zn) \cdot m(Zn) + c_p(Cu) \cdot m(Cu)] \cdot \Delta T$

$Q_p = -(4{,}19 \cdot 4{,}0 + 0{,}389 \cdot 1{,}069 + 0{,}385 \cdot 0{,}127)\ J/K \cdot 23\ K = \underline{\underline{-396\ J}}$

c_p: spec.capacity of heat at 20 °C
m(Cu): Mass of copper
m(Zn): Mass of zinc minus oxidized part
$c(Zn) = 0{,}389\ J \cdot g^{-1} \cdot K^{-1}$
$c(Cu) = 0{,}385\ J \cdot g^{-1} \cdot K^{-1}$
$c(H_2O) = 4{,}19\ J \cdot g^{-1} \cdot K^{-1}$

$n_0(Zn) = m(Zn) / M(Zn) = 1.2\ g / 6.39\ g \cdot mol^{-1} = 0.01835\ mol$
$n(Cu) = c(CuSO_4) \cdot V_L = 0{,}5\ mol/L \cdot 0{,}004\ L = 0{,}002\ mol$
$n(Zn) = 0{,}018\ mol - 0{,}002\ mol = 0{,}016\ mol$

$m(Cu) = 0{,}002\ mol \cdot 63{,}546\ g\ mol^{-1} = 0{,}127\ g$
$m(Zn) = 0{,}016\ mol \cdot 65{,}39\ g mol^{-1} = 1{,}069\ g$

Calculation of the molar change of enthalpy:

$$\Delta_{Rm}H = \frac{Q}{n(Cu)} = \frac{-396J}{0{,}002mol} = -198\frac{kJ}{mol}$$

Reference: $\Delta_{Rm}H = -216{,}96$ kJ/mol

3. A no-cost piezoelectric device (PED) made from an empty cigarette lighter (D. Giesler)

A piezoelectric device produces voltages of thousands of volts resulting a spark between two electrodes igniting for instance the gas of a cigarette lighter. In MCE such a spark is needed for a volumetric water synthesis from a stoichiometric mixture of H_2 and O_2.

Assembling a *low-cost PED* needs a syringe and a PED available in shops for electronic materials.

The following chart demonstrates how to make gives an idea to make how an empty disposable lighter can be re-used to make a no-cost PED.

- You need materials for welding, sand paper, 1 insulated wire with 2 crocodile clips
- Cut the cable, remove 3 mm of the insulation
- Sandpaper the two contacts of the piezoelectric part of the lighter
- Weld the ends of the cables like shown in Fig. 11.
- Re-assemble the lighter-PED (Fig. 12) to have a flexible tool for the experiment 4.1 in the following article**.**

For detailed informations look for the MCE book published by the Arab Academic College for Education in Haifa (muha4@macam.ac.il).

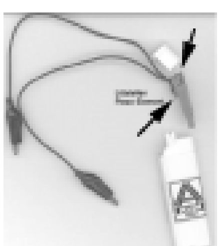


Figure 11
Parts of lighter after welding with two wires ending in crocodile clips.

Figure 12
Lighter PED after reassembling ready for use (Fig. 13.1.3. + 4).

4. Volumetric electrolyses of Glauber salt (M.K. El-Marsafy, M. Hugerat, P. Schwarz)

4.1. Electrolysis of Glauber Salt in two barrel pipettes followed by a volumetric synthesis

Many methods for the *volumetric water analysis* have been developed since the German chemist A. W. von Hofmann (1803 – 1892) constructed his apparatus for the electrolysis of water. In Fig. 13 the glassware is replaced by two barrel pipettes dipping into a 50mL infusion bottle. The pipettes and the bottle contain sodium sulfate solution + a drop of universal indicator. The platinum electrodes are replaced by hypodermic needles (size 17). A 9 Volt battery is used as power supply. A photovoltaic solar panel (6 or 12 V) is also suitable. After reading the volumes of the gases in the cathode and anode half cells (Fig. 14) the polarity is changed until the volumes in the heads of both pipettes are equal (Fig. 15, left). Each of the two pipettes will then be transformed into eudiometers for *volumetric water synthesis*. (Fig. 15 right, Fig. 16) The gas mixture is ignited using the piezoelectric device described in the previous article (Figs. 11-12).

The strong reduction of volume is explained by the fact that the oxy hydrogen gas is transformed into liquid water.

Figure 13
Pipette Hofmann 1:
Na_2SO_4(aq) + indicator in 2 barrel pipettes pierced by electrodes and dipping in a bottle as salt bridge.

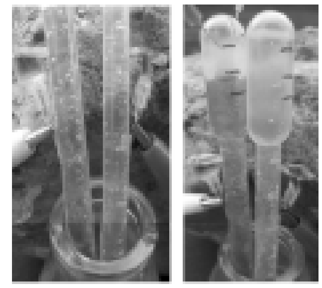


Figure 14
Pipette Hofmann 2:
Red: $2\ H_2 + 2e^- \rightarrow H_2(g + 2\ OH^-(aq)$
ox: $H_2O \rightarrow ½\ O_2(g) + 2\ e^- + 2\ H^+\ (aq)$

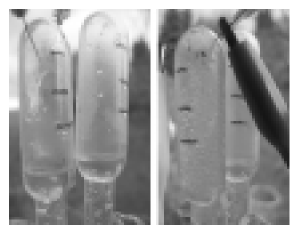


Figure 15
Left: Result of changing the polarity
Riggt: Volumetric water synthesis 1

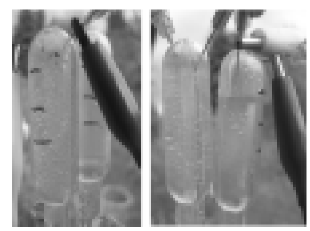


Figure 16
Volumetric water synthesis 2
(in the right pipette head):
$2\ H_2(g) + ½\ O_2(g) \rightarrow H_2O(l)$

4.2. Electrolysis of Glauber Salt in two Liquemin injection bottles

- Make 30 mL of red cabbage extract by boiling red cabbage in water and decanting the blue supernatant.
- Add a teaspoon full of Glauber Salt (Na_2SO_4), stir to get a solution.
- Cut off the bottoms of two disposable drinking beakers (Fig. 17).
- Pierce one of them from downside by two blunted injection needles. (Distance between the needles 2 cm).
- Totally fill two 5mL Liquemin injection bottles with the electrolyte, add 3 mL of the electrolyte to the pierced container.

Figure 17
Bottle Hofmann 1:
Na_2SO_4(aq) + red cabbage juice

Figure 18
Bottle Hofmann 2:
red: $2\ H_20 + 2\ e^- \rightarrow H_2(g) + 2\ 0H^-(aq)$
ox: $H_20 \rightarrow ½\ O_2(g) + 2\ e^- + 2\ H^+(aq)$

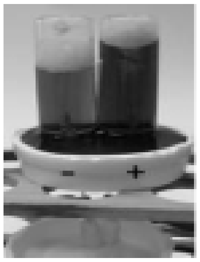


Figure 19
Bottle Hofmann 3:
red: $2\,H_2O + 2\,e^- \rightarrow H_2(g) + 2\,OH^-(aq)$
ox: $H_2O \rightarrow \frac{1}{2}\,O_2(g) + 2\,e^- + 2\,H^+(aq)$

Figure 20
Bottle Hofmann 4:
red: $2\,H_2O + 2\,e^- \rightarrow H_2(g) + 2\,OH^-(aq)$
ox: $H_2O \rightarrow \frac{1}{2}\,O_2(g) + 2\,e^- + 2\,H^+(aq)$

Connect the needle electrodes to a 9-Volt battery.

Testing for hydrogen, getting more oxygen and testing for oxygen

- Remove the left bottle from its electrode, lit a candle.
- Try a negative oxy hydrogen test like seen in Fig. 21.
- Go on with the electrolysis until the right bottle is full of gas.
- Do a "glowing tissue paper test for oxygen (Fig. 22).

Figure 21
Left bottle Hydrogen-Test:
$\mathbf{H_2(g) + \frac{1}{2}\,O_2(g) \rightarrow H_2O(l)}$

Figure 22
Right bottle Oxygen-Test:
$C(s) + O_2(g) \rightarrow CO_2(g)$

4.3. Electrolysis of Glauber Salt on a white plastic surface

Less is more: In two minutes you can not only do the *redox reaction* of water electrolysis (Glauber Salt can even be replaced by tap water) but also the *acid base reaction* of neutralization by mixing the basic (yellow) and acidic (red) areas around the electrodes.

- For the electrolysis of Glauber Salt on a plastic surface pierce a white film canister by 2 sterile small hypodermic injection needles 5-10 mm apart.
- Take a big drop of Na_2SO_4 solution in red cabbage juice extract (Fig. 23).
- Connect the needles with the two terminals of a 9-Volt-battery.
- Observe the result (Fig. 24).

Figure 23
Electrolysis in a drop 1:
Materials and chemicals

Figure 24
Electrolysis in a drop 2:
red: $2\,H_2O + 2\,e^- \rightarrow H_2(g + 2\,OH^-(aq)$
ox: $H_2O \rightarrow \frac{1}{2}\,O_2(g) + 2\,e^- + 2\,H^+(aq)$

- Remove the needles and mix the areas of changed colours using a piece of copper wire as a stirring rod.

$$2H^+_{(aq)} + 2OH^-_{(aq)} \rightarrow 2\,H_2O_{(l)}$$

"2 + 2 = 3"

5. Microscale Experiment on Volume Reduction during Mixing

Introduction

Most of the high school students are not aware that the chemistry problems differ from mathematical ones and if you ask them: What will be the volume of the solution prepared from 50 mL water and 50 mL ethanol the answer will be almost always: 100 mL. This only proves that they know elementary math and that they observed this system only macroscopically, like setting domino blocks and the length will be a sum of the length of each piece. This would work if the matter was a continuum consisting of a system "block" and not of tiny particles each with individual properties.

The proposed experiment may easily clarify some misunderstandings in the properties of matter. This experiment proves that the matter is not a continuum but a system consisting of individual particles that are interacting each other.

This experiment performed on macro scale level proves that after mixing the total volume is reduced by approximately 4%.

Materials:

2 test tubes (4 mL), test tube stand, small piece of glass or a glass grain, rubber stopper that fits on the test tube hole, capillary, Pasteur pipettes, gloves.

Chemicals:

Ethanol, paraffin oil, distilled water.

Experimental

- Transfer water into one 3mL test tube and ethanol into the other one.
- Add some food dye and mix.
- With a pipette put 1.5 mL of the coloured water in a third test tube.
- Add a piece of glass.
- With another pipette add few drops of paraffin oil to separate the water from the ethanol.
- Now carefully transfer 1.5 mL of coloured ethanol with a dry pipette.
- Close the test tube with a rubber stopper that has a capillary as shown in Fig. 25.
- By pressing the stopper provide the capillary with a high level of coloured ethanol.
- Cut a glove and put a piece on the forefinger.
- Invite the students to watch and mark the level of ethanol with permanent marker before mixing (Fig. 26).
- Close the upper hole of the capillary with the finger and mix the three liquids by tilting twice the test tube for more than 90 degrees (Fig. 27).
- After that ask for attention on the capillary and remove your finger.
- Mark the new level (Fig. 28). For quantitative evaluation measure the inner diameter of the capillary and calculate the difference of the two volumes.

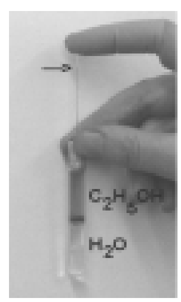


Figure 25
Ethanol and water before mixing.

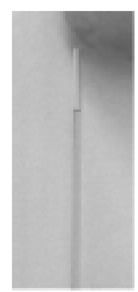


Figure 26
Level of ethanol in the capillary.

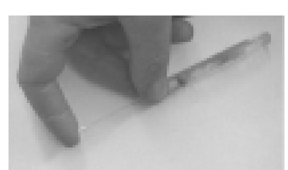


Figure 27
Mixing ethanol and water.

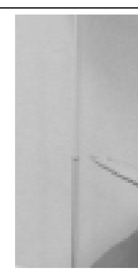


Figure 28
Level in the capillary after mixing.

Observation and discussion

Due to the small diameter of the capillary a spectacular volume reduction is visualized with a small quantity of the two liquids.

The volume reduction is a result of a re-arrangement of the two different molecules and their hydrogen bonding. In other words, molecules of water and of ethanol are packed in a certain way, and upon mixing they are repacked and the experiment shows that these molecules are much better packed when they are together in mixture.

Notes

The glass piece is used to provide better mixing and the food colours are applied to make this phenomenon more vivid.

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Estrategia de motivación para el aprendizaje de los estudiantes de educación superior
Motivation strategy for learning in higher education students

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Resumen

La falta de interés de los estudiantes en varias áreas dentro de la licenciatura de ingeniería civil es una situación que se ha presentado desde hace mucho tiempo y que por lo mismo se ha convertido en una mala costumbre dentro del ambiente estudiantil; situación que afecta decisivamente en la conclusión de la licenciatura y aún más en la consideración de realizar estudios de posgrado y consecuentemente, de investigación. Por lo anterior, este trabajo presenta el caso específico y muy común de la materia de Mecánica de Suelos, asignatura que, aunque es indispensable como muchas otras, no resulta ser del agrado de los estudiantes y terminan acreditándola de forma apática y desinteresada, lo que ocasiona bajos rendimientos y que los alumnos olviden gradualmente o hasta inmediatamente todo su contenido. Consecuentemente, surge la necesidad de aplicar e innovar alguna estrategia que pueda conseguir la obtención de un mejor rendimiento en los alumnos, e implícitamente, la motivación requerida para realizar satisfactoriamente sus estudios de licenciatura y generar el deseo de estudiar un posgrado e investigación.

Palabras clave: educación superior, educación en ingeniería, aprendizaje en ingeniería, motivación educativa.

Abstract

The lack of interest of the students in several areas at the undergraduate level of civil engineering is a situation that has been present for quite a while, and for the same reason it has turned into a bad habit within the student environment. It affects decisively the conclusion of the baccalaureate, and has an even worse effect on the undertaking of graduate studies and thus, research. Because of the above, this work presents the specific and very common case of the soil mechanics courses, a subject that, although it is as indispensable as the others, does not turn out to give students pleasure, and they end up taking it in an apathetic and uninterested manner. This causes low achievement levels and students gradually or even immediately forget all of its content. Therefore, the need has arisen to apply and introduce some strategy that could obtain a better learning levels from the students and implicitly, the motivation needed to achieve their baccalaureates satisfactorily, along with the desire to pursue graduate studies and research.

Key words: higher education, engineering education, engineering learning, educational motivation

INTRODUCCIÓN

El continuo desinterés por el área de ingeniería civil y en este caso por la asignatura de Mecánica de Suelos se ha venido presentando en los estudiantes desde siempre, acentuándose en los últimos años, esta situación ha provocado gran preocupación en los especialistas del área por la diversidad de problemas en la materia (investigación y práctica) que han estado y siguen siempre pendientes por investigar y resolver, debido a que no se cuenta con los suficientes recursos humanos capacitados para ello. Esta preocupación generalizada ha originado el planteamiento de algunas estrategias que pudieran ayudar a canalizar más estudiantes hacia esta área.

METODOLOGÍA

La metodología consistió en la realización de encuestas para analizar y detectar el origen del problema, que se concentraba principalmente en la falta de interés de una materia y por ende, el bajo rendimiento, para luego aplicar una estrategia al grupo de estudiantes y observar cuál había sido la mejora.

Origen del problema

Una gran variedad de causas de todos los tipos que justificaran la tan manifestada falta de interés en la materia, pudiera existir, y no sólo en ésta en específico, sino en cualquier otra.

El aumento de la edad y de la escolaridad muestra una relación positiva en el desarrollo del razonamiento moral de principios, pues los sujetos con escolaridad de maestría aventajan en todas las variables a los otros grupos (BARBA, 2002).

De antemano se tienen antecedentes que los problemas más comunes en este tipo de licenciatura siempre han sido los económicos y, en consecuencia, los relativos a la alimentación y nutrición del individuo. Se realizó una encuesta en un grupo de alumnos de trabajando y estudiando simultáneamente, lo que contribuye en gran medida en la disminución del rendimiento de los estudiantes por la falta de concentración, exceso de cansancio y de sueño que se presenta normalmente como consecuencia de lo anterior. Respecto a la alimentación, el 70% de los integrantes de este grupo, asisten a clase sin haber desayunado lo necesario para estar por períodos de hasta nueve horas continuas recibiendo clases. De ahí, las siguientes comidas son incompletas y raquíticas, lo que hace disminuir el hambre sin satisfacer completamente la necesidad fisiológica y, mucho menos, el aspecto nutricional.

Por otro lado, se encuentran los problemas de tipo vocacional acompañados por un mal manejo de los métodos de estudio que ocasionan falta de interés y bajo rendimiento. Este problema se origina desde los estudios medios superiores, ya que no se vigila estrictamente este aspecto tan importante para el desarrollo futuro de los adolescentes en su vida