C.J. du Toit, S.J. Nel, G. Lachmann, J.J.A. Smit and J. Labuschagne,
S. Afr. J. Chem., 2004, **57**, 19–23,
<http://journals.sabinet.co.za/sajchem/>.

An Investigation into the Use of Microchemistry Experiments in First-year University-level Practicals

Cornelius J. du Toit*, Stephanus J. Nel, Gerhard Lachmann, Jan J.A. Smit and Johan Labuschagne

School of Chemistry and Biochemistry, North West University, Potchefstroom Campus (PUK), Potchefstroom, 2520 South Africa.

Received 19 January 2000; revised 4 February 2002; accepted 10 January 2003.

ABSTRACT

In the case study reported on in this article the desirability to convert first-year university-level practicals in chemistry from conventional techniques to microscale methods was investigated. The results indicated that microscale experiments did not result in a noticeable saving in time and gave results that compare favourably with those of the conventional experiments. The most positive aspect of microscale experiments is a long-term financial one. A few negative aspects of microscale experiments were identified in the study.

OPSOMMING

In hierdie bydrae word 'n gevallestudie gerapporteer oor die wenslikheid om eerstevlak universiteitschemie-prakties van konvensionele tegnieke na mikroskaaltegnieke om te skakel. Die resultate toon dat die mikroskaaleksperimente nie noemenswaardig tydsbesparend is nie en resultate lewer wat goed vergelyk met dié van konvensionele eksperimente. Die mees positiewe aspek van mikroskaaleksperimente is finansiële besparing op die lang termyn. Enkele negatiewe aspekte van mikroskaaleksperimente is in die ondersoek geïdentifiseer.

KEYWORDS: **microscale, practicals, chemicals, Microchemistry, first-level.**

1. Introduction

At present, the science education community accepts that the most effective way students learn science is through active involvement in experimental work.[1] Experimental work is, however, very expensive and with budgets of departments in the science faculties of universities under constant pressure, the conventional practical classes involving large groups of first-year students can hardly be afforded anymore. The utilization of microchemistry sets may provide a solution to the problem. Questions to be addressed when considering a change from the conventional use of large-scale apparatus and large amounts of chemicals to the microscale are:

- How do students experience the microchemistry approach compared to that of the conventional approach?
- How cost-effective is microchemistry?

These questions were investigated in the study reported in this article.

2. Microscale Experimenting

The microscale experimental approach developed rapidly during the past decade in countries like the USA, the UK and Australia.[2–7] In the microscale approach very small amounts of chemicals (milligrams of a solid, a few drops of liquid, a single crystal) are used in specially constructed well-plates normally used in biochemistry or microbiology.

According to McGuire *et al.*[8] the implementation of this approach was initiated by a series of articles in 1985 in the *Journal of Chemical Education.* Microscale experimentation then not only took root in universities in the USA and Britain, but was also implemented in schools in these countries.

The rapid growth in popularity of the microscale experimentation method can be attributed to factors like safety and waste regulations in developed countries, the high cost of conventional laboratories and availability and high cost of chemicals.[9–12]

* To whom correspondence should be addressed.
E-mail: checjdt@puk.ac.za

3. Survey

The study reported in this article was conducted at the North West University, Potchefstroom Campus (PUK), South Africa, as part of the first-year chemistry practical teaching. The population of students subjected to this study was a group of 480 enrolled for Chemistry 1. The authors were allowed to modify some of the practicals to microchemistry procedures for one of the weekly practical sessions. For the purpose of the survey, the weekly practical sessions attended by students following a substantially chemistry-orientated study course (B.Sc., B.Eng.) were selected. This comprised 50 students who were divided at random into two groups of 25 students each, an experimental group and a control group. In order to cause least disruption to the course, the experimental group was subjected to only four microscale experiments while the control group performed these experiments in the conventional way. The rest of the group (430) conducted the experiments in the conventional way. All 480 students completed the questionnaires that formed part of the study. The four experiments converted to microscale were selected to cover as wide range as possible of educational objectives and activities. The experiments included qualitative and quantitative aspects of practical chemistry as well as experiments aimed at developing certain skills (e.g. titration) in first-year practical courses.

One of the authors presented the practicals to both the experimental and control groups. Laboratory assistants were briefed on the microscale as well as the traditional experiments.

The four experiments selected for this study were:

- the synthesis of Nylon-6,10;
- an investigation of the reactions of strong and weak acids with strong and weak bases by using different indicators;
- the percentage purity of calcium carbonate; and
- the preparation of lead chloride and lead chromate from lead carbonate.

Worksheets to be completed by students were supplied with each experiment. The worksheets used by the experimental group were the same as used by the control group with as little as possible modifications to accommodate the microscale techniques to minimize any possible bias.

Information on students' attitudes, skills and knowledge was gathered by means of interviews and observation while they were performing experiments. A pre-survey questionnaire was administered at the beginning of the semester, before the practicals started, and a post-survey questionnaire was administered at the end of the semester, after the last practical session had been completed. Students in the experimental and control groups were asked to answer a few questions in writing concerning the experiment conducted as a complement to the interviews at the end of every practical session.

Apparatuses used for the microscale experiments included inexpensive plastic well-plates of the type frequently used in research in the life sciences. Plastic tube pipettes were converted into pipettes able to deliver small, medium and large drops, micro weighing boats, spatulas and stirrers.[3] Both the micro well-plates and apparatuses made from tube pipettes are hydrophobic and thus easy to clean. Other apparatuses, easily constructed by the students, were also used.

4. Results

4.1. Exposure to Practical Work at School Level

Prior to the first practical session, all 480 students completed a pre-survey questionnaire aimed at a situation analysis. This revealed that 26% of all first-year students had never performed experimental work in Grades 11 and 12 at school. Nineteen per cent had performed experiments once a month and 24% once every quarter. Seven of the experimental group of 25 students never did any practical work at school, seven did so once in three months and another seven did practical work in chemistry once a month. Of the control group of 25 students, 12 never did any practical work at school, one did so once in three months and another eight did practical work once a month.

From the situation analysis it was evident that the factors that had a positive effect on the students' interest in chemistry in Grades 10–12 were demonstration experiments (82%), hands-on practical work (76%), career opportunities (57%) and the science teacher (56%). This information regarding the impact of practical work on interest (attitude) was further motivation to examine the effect of practical work, and especially microscale practical work at first-year university level.

4.2. Attitude towards Practical Work — Start and End of Semester

The responses of the experimental and control groups with regard to the items directed at determining attitude towards practical work in the pre- and post-survey questionnaires are given in Table 1.

4.3. Results Obtained from the Four Experiments

4.3.1. Experiment 1. The Synthesis of Nylon-6,10

The objective was to produce a nylon thread. Since aggressive organic solvents were used, a porcelain crucible lid replaced the well-plate. This was done because the well-plate is not inert to all organic solvents. Both the experimental and control groups had problems in producing a thread at the first attempt, but were successful during a second attempt. The experimental (microscale) group used between five and 10 times less of the chemicals in completing the experiment than the control group. The microscale group also spilled less of the chemicals on the benches than the control group. The duration of the microscale experiment was not significantly longer (on average seven minutes) than the conventional one. The experimental group indicated that they could clearly observe what was happening.

4.3.2. Experiment 2. The Reactions of Strong and Weak Acids with Strong and Weak Bases using a Variety of Indicators.

The assignment required a number of titrations to investigate the suitability of a range of indicators for the different types of titrations. In this experiment the control group worked in pairs while the microscale group performed the experiments individually since well-plates were used. On average, the experimental group completed their experiments 10–25% faster than the students in the control group. The experimental group, however, could not observe the colour changes clearly in all reactions because the well-plates had a bluish tint.

4.3.3. Experiment 3. Determination of the Percentage Purity of Calcium Carbonate

Three different samples of substances containing calcium carbonate were supplied to the students, namely ostrich eggshell, seashells and pure calcium carbonate. Owing to the slow reaction of diluted hydrochloric acid with calcium carbonate, more than half of all students were concerned that they would not be able to complete the experiment on time, and consequently started working faster, which influenced their results negatively.

This experiment is an example of the back titration technique. The complexity of the calculations influenced both groups. This was apparent from the large number of students who requested help from the lecturer and laboratory assistants. The time taken to do the calculations also reflected the degree of difficulty. Most students completed the experimental part in 90 minutes, but another 90 minutes was spent on the calculations.

In the experimental group, four students were not successful in determining the percentage purity of their samples within the allotted time compared with three in the control group. The group that performed the microscale experiment indicated that they experienced it as difficult and complicated. The experiment required a high level of concentration and precision work. The experimental group experienced no problems observing the colour changes at the end point of the titration.

4.3.4. Experiment 4. Preparation of Lead Chloride and Lead Chromate from Lead Carbonate

A range of experimental techniques was used during these syntheses that are difficult to adapt to microscale experimentation. It was difficult to determine when a slight excess of precipitating reagent had been added owing to the small volume of the wells in the well-plate. The technique to remove excessive water from the crystals in the wells was also much more difficult to master than the simple filtration that was used by the control group. Both groups experienced the calculations as being difficult. Students in the experimental group in general did not obtain good results in their first attempts at carrying out the experiment. Eight of the students that performed the microscale experiments obtained good results on the first attempt. In the control group, only two students were unsuccessful on the first attempt. Most students in the microscale group whose first attempt was unsuccessful, repeated the experiment successfully in a much shorter time than in the initial attempt, indicating that a little exercise improves a student's experimental skills considerably. The time to complete the microscale experiment, including the

Table 1 Attitude towards chemistry practicals, and change in attitude during the first semester.

Item	Agree (%)	Undecided (%)	Disagree (%)
Pre-survey questionnaire			
(1) Item not included			
Post-survey questionnaire			
(1) My attitude towards chemistry practicals is negative			
Total group	22	19	59
Experimental group	11	22	67
Control group	17	17	66
Pre-survey questionnaire			
(2) I prefer doing practicals to attending lectures			
Total group	66	20	14
Experimental group	84	5	11
Control group	70	25	5
Post-survey questionnaire			
(2) I prefer doing practicals to attending lectures			
Total group	54	27	19
Experimental group	61	33	6
Control group	65	35	0
Pre-survey questionnaire			
(3) I prefer doing practicals to watching demonstrations			
Total group	68	17	15
Experimental group	84	11	5
Control group	85	10	5
Post-survey questionnaire			
(3) I prefer doing practicals to watching demonstrations			
Total group	63	21	16
Experimental group	78	5	17
Control group	76	12	12
Pre-survey questionnaire			
(4) At school more time must be allocated for practicals			
Total group	26	39	35
Experimental group	84	16	0
Control group	95	5	0
Post-survey questionnaire			
(4) In CHE111 more time must be allocated for practicals			
Total group	21	39	40
Experimental group	28	44	28
Control group	18	12	70
Pre-survey questionnaire			
(5) At school I preferred reading my textbook to doing practicals			
Total group	12	18	70
Experimental group	5	5	90
Control group	5	3	65
Post-survey questionnaire			
(5) In CHE111 I prefer reading my textbook to doing practicals			
Total group	14	22	64
Experimental group	5	17	78
Control group	18	12	70
Pre-survey questionnaire			
(6) The more time I spent on practicals at school the more my interest in chemistry grew			
Total group	50	30	20
Experimental group	67	28	5
Control group	52	38	10
Post-survey questionnaire			
(6) The more time I spent on practicals at university the more my interest in chemistry grew			
Total group	38	33	19
Experimental group	67	16	17
Control group	50	28	22

Continued on p. 22

Table 1 (*continued*)

Item	Agree (%)	Undecided (%)	Disagree (%)
Pre-survey questionnaire			
(7) My interest in chemistry declines if I do not do practicals			
Total group	40	33	27
Experimental group	61	22	17
Control group	24	43	33
Post-survey questionnaire			
(7) My interest in chemistry declines if I do not do practicals			
Total group	38	33	29
Experimental group	44	17	39
Control group	28	33	39
Pre-survey questionnaire			
(8) Chemistry practicals at school were boring			
Total group	16	16	68
Experimental group	17	5	78
Control group	14	29	57
Post-survey questionnaire			
(8) Chemistry practicals at university were boring			
Total group	18	23	59
Experimental group	17	11	72
Control group	17	22	61
Pre-survey questionnaire			
(9) Chemistry practicals at school were a waste of time			
Total group	10	19	71
Experimental group	0	6	94
Control group	0	38	62
Post-survey questionnaire			
(9) Chemistry practicals at university were a waste of time			
Total group	12	23	65
Experimental group	11	6	83
Control group	28	50	22

second effort, was 125 minutes on average, while in the conventional way it took on average 117 minutes. From personal interviews it was evident that both groups were confident that they had completed the experiment successfully.

4.4. Comparison of Costs (2001 Prices)

In Table 2 the cost of the conventional experiments is compared to the cost of the microscale experiments. As the same apparatuses are used for many experiments, only the total apparatus cost is given.

5. Discussion of Results

First-year chemistry students generally have a positive attitude towards chemistry practicals (Table 1, pre-survey, item's 2, 3, 5, 8 and 9). Results from the post-survey indicate that there was a decline in enthusiasm for practical chemistry (Table 1, post-survey items 2, 3, 5, 8 and 9). This is in agreement with results of an earlier survey.[13]

The results given in Table 1 do, however, indicate that the responses of the experimental group, the 25 students who did the four experiments converted to microscale, differ from those of the other students. They were more positive (or considered from a different perspective, less negative) than those of the whole group of 480 students. Their responses were also less negative than those of the control group of 25 students who did the conventional experiments.

A comparison of the results obtained from the experimental and control groups suggest that:

- The microscale experiments did not result in a noticeable saving of time.
- The microscale experiments were not experienced as being easier than the conventional ones.
- The microscale experiments were noted in some cases as being problematic because the students could not observe the physical changes clearly.
- In some of the microscale experiments the students experienced difficulties in achieving the required results, but succeeded during a second attempt and within the allotted time for the practical session.
- The microscale experiments delivered results which compared favourably with those of the conventional experiments. Both with regard to quantitative and qualitative experiments, the results were within acceptable limits for experimental errors.
- Lecturers and student supervisors need to be trained properly in the handling of microscale apparatuses. Their lack of skill may be a negative factor in the microscale laboratory.
- The saving in chemicals and waste disposal is estimated to be R3.42 per student, which is much less than the cost of acquiring microscale apparatus.

6. Conclusion

The observed influence of microscale methods had an effect on the attitude that was not large enough to be taken into account in

Table 2 Comparison of costs (2001 prices).

Experiment	Conventional cost/student (R)	Microscale cost/student (R)
Experiment 1		
The synthesis of Nylon-6,10		
Reagents	5.04	3.79
Reagent disposal	0.96	0.48
Experiment 2		
An investigation into the reactions of strong and weak acids with strong and weak bases with the aid of a variety of indicators.		
Reagents	0.08	0.02
Reagent disposal	0.00	0.00
Experiment 3		
Determination of the percentage purity of calcium carbonate.		
Reagents	0.10	0.04
Reagent disposal	0.00	0.00
Experiment 4		
Preparation of lead chloride and lead chromate from lead carbonate.		
Reagents	2.06	0.73
Reagent disposal	0.48	0.24
Total reagents	8.72	5.30
Apparatus	408.04	38.04
Total cost	416.76	43.34

this case study. The quantitative and qualitative results of the microscale experiments compare favourably with that of the conventional experiments. The financial gain often mentioned in the literature is based on the low cost of chemicals and waste disposal of microscale experiments. For experiments in which low cost and less hazardous chemicals are used the low consumption of chemicals poses no real incentive to use microscale techniques. No short-term financial benefit is attained by converting established conventional laboratories to microscale techniques, but in the case of the establishment of a new laboratory substantial savings could be made if microscale techniques were to be adopted from the beginning.

Negative factors of microscale methods are restricted visibility of some microscale reactions, the reaction of the well-plate with certain organic solvents and the difficulty in heating the content of the wells in the well-plates.

References

1. P. Dechsri, L. Jones and W.L. Hekkinen, *J. Res. Sci. Teaching,* 1997, **34**, 891.
2. S.W. Breuer, *Educ. Chem.*, 1991, 75.
3. S. Thomson, *Chemtrek: Small-scale Experiments for General Chemistry,* Allyn and Bacon, Massachusetts, 1990.
4. Z. Szafran, M.M. Singh and R.M. Pike, 1989, *J. Chem. Educ.,* **66**, A263.
5. K.L Williamson, *Am. Lab.,* 1991, 48.
6. W.F. Beasley, *Austr. Sci. Teachers. J.,* 1993, **39**, 50.
7. J. Crump and R.K. Sandwick, *J. Chem. Educ.,* 1994, **71**, A199.
8. P. McGuire, J. Ealy and M. Pickering, *J. Chem. Educ.,* 1991, **68**, 869.
9. C.A. Bennett, J.J. Mouch, F. Strivers and E.K. Melton, *J. Chem. Educ.,* 1989, **66**, A90.
10. M. Chloupek-McGough, *J. Chem. Educ.,* 1989, **66**, 92.
11. M. Crosson and R. Gibb, *J. Chem. Educ.,* 1992, **96**, 830.
12. A.P. Zipp, *J.Chem. Educ.,* 1989, **66**, 956.
13. M.M. de V. Steyn, C.J. du Toit and G. Lachmann, *S. Afr. J. Chem.*, 1999, **52**