# Green Chemistry at the Microscale: Driving Sustainability Consciousness and Engagement in Higher Education

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**Abstract:**
Green Chemistry refers to the design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances and integrates sustainability into the fundamental design of the chemical processes. The intellectual and philosophical foundation of green chemistry was laid down by Paul Anastas and John Warner in the form of twelve principles of green chemistry in 1998. The Green Analytical Chemistry (GAC) emerged from these twelve principles and applies them to analytical methods. For smooth application of these principles, they were revised in 2013 by Gałuszka, Migaszewski, and Namieśnik in the form of SIGNIFICANCE mnemonic so that they could be easily applied in the analytical processes being carried out in the laboratories at every level. The present study has been carried out for a period of three years using microscale analytical techniques namely spot analysis, double burette titration and Middleton method. The main focus was on exploring student interest and sustainability consciousness by generating little waste, increased safety for operator and eliminating toxic reagents while carrying out the experiments in the chemistry laboratory. The students' responses were assessed through semi structured interviews, questionnaires and observation of laboratory practices. The results demonstrated that these microscale techniques heightened interest and engagement in laboratory work amongst the students by 60% and increased sustainability awareness by 65%. The study concludes that incorporating microscale techniques of GAC in laboratories of higher education institutes can enhance students' engagement with interest in laboratory work and increase ethical responsibility for sustainable practices.

**Keywords:** Green Analytical Chemistry, sustainability, microscale.

## Introduction:

Green Chemistry is not just a branch of chemistry but it is discipline which covers all the fields of chemistry and specifically the analytical branch of chemistry. It was initially defined as the design of such products and processes which minimize the use and generation of hazardous substances and was based on the 12 principles of green chemistry (Table 1) by Paul Anastas and John Warner in 1998. "[1,2]"

**Table 1: The twelve Green Chemistry Principles**

| Green Chemistry Principle | How to Implement in Chemistry Laboratories |
|---|---|
| Principle 1: Waste Prevention | Plan to avoid generation of waste |
| Principle 2: Atom Economy | Reduce waste at molecular level |

| | |
|---|---|
| Principle 3: Less Hazardous Chemical Synthesis | Doing synthesis of compounds using less hazardous chemicals |
| Principle 4: Designing safer Chemicals | Minimize toxicity directly by molecular design |
| Principle 5: Safer solvents and auxiliaries | Choose the safest solvent available for a given step |
| Principle 6: Design for energy efficiency | Choose the least energy intensive route |
| Principle 7: Use of renewable feedstock | Use chemicals made from renewable sources |
| Principle 8: Reduce derivatives | Minimize the use of derivatives |
| Principle 9: Catalysis | Minimize the use of stoichiometric reagents and use catalysts preferably |
| Principle 10: Design for degradation | Design chemicals that can be easily discarded and do not bioaccumulate |
| Principle 11: Real time Pollution Prevention | Monitor chemical reactions in real time as they occur to prevent release of hazardous substances in the environment. |
| Principle 12: Inherently safer Chemistry for Accident Prevention | Choose procedures that are inherently safer and minimize the risk of accidents |

GAC emerged from green chemistry in 2000. This relatively new area of activity within green chemistry concerns the role of analytical chemists in making laboratory practices simpler, cost effective and environmentally friendly while preferring microscale, operator friendly procedures. "[3]" Singapore: Springer Singapore. Green Analytical chemistry also fulfils the goals of sustainable chemistry (SC) a term which encompasses use of efficient, effective, safe and environment friendly benign chemical products and processes. "[4,5]"

GAC focusses on Elimination or reduction of the use of chemical substances (solvents, reagents, preservatives, additives for pH adjustment and others), minimization of energy consumption, proper management of analytical waste and increased safety for the operator.The twelve revised principles of GAC under the mnemonic SIGNIFICANCE given by Gałuszka, Migaszewski, and Namieśnik consider different aspects of the analytical methods and suggest greenest solutions for each procedure followed in the laboratories (Table 2). "[6]" Various studies have been done through various tools and metrics on the greenness of procedures adopted by laboratories using these Principles. "[7]". The microscale analytical techniques which utilize minimum chemicals, less cumbersome apparatus can be adopted in the chemistry laboratories in the undergraduate and postgraduate levels to promote safe, student friendly experiments which are less time consuming and allow miniaturization in all respects. "[8]"

**Table 2: Revised Green Analytical Principles: SIGNIFICANCE Mnemonic**

| **S.No.** | **"SIGNIFICANCE" Mnemonic** | **REVISED GREEN CHEMISTRY PRINCIPLES** |
|---|---|---|
| | S | Select direct analytical technique. |
| | I | Integrate analytical processes and operations. |
| | G | Generate as little waste as possible and treat it properly. |
| | N | Never waste energy |
| | I | Implement automation and miniaturization of methods. |

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| | | |
|---|---|---|
| | F | Favor reagents obtained from renewable source. |
| | I | Increase safety for operator |
| | C | Carry out in-situ measurements. |
| | A | Avoid derivatization. |
| | N | Note that the sample number and size should be minimal. |
| | C | Choose multi-analyte of multi-parameter method. |
| | E | Eliminate or replace toxic reagents. |

**Objectives of the study:**

The main objective of this study has been to explore students enhanced engagement in chemistry and sustainability consciousness through practical introduction of microscale techniques of GAC which can be easily carried out in the laboratories of higher education institutes offering chemistry as a subject. "[9, 10]" Hence the main objectives of the study are:

1. To introduce the microscale green analytical techniques like double burette method for volumetric analysis, spot method for inorganic analysis and Middleton method for organic elemental analysis amongst the Undergraduate and Postgraduate students studying chemistry as major subject.
2. To know about the enhanced engagement students with interest in chemistry practical through safer microscale techniques.
3. To find the role of microscale techniques in enhancing sustainability consciousness of the students beyond laboratories.

**Methodology**:

The present study adopted both quantitative and qualitative approaches to comprehensively assess the impact of green microscale analytical techniques on students' interest in chemistry and effect on their sustainability consciousness. The study sample comprised 52 undergraduate and postgraduate students of Chemigreen club of Department of Chemistry of Shree Guru Gobind Singh Ji Government College (SGGSJ GC) Paonta Sahib, Himachal Pradesh. Informed consent was obtained from all respondents in the beginning of academic session.

The experiments chosen for the present study included volumetric analysis, inorganic mixture analysis and elemental organic analysis. (Table 3) During the various practical sessions, the students performed the experiments through three green analytical microscale techniques and five main revised principles considered for the present study were taken from the SIGNIFICANCE mnemonics. (Table 2, S. No. (3), (5), (7), (10), (12)

**Table 3: Experiments performed with Green Analytical Microscale Techniques**

| S.N | Experiments considered | Conventional method | Green analytical microscale technique followed | Revised Green Principle from SIGNIFICANCE Mnemonic |
|---|---|---|---|---|
| **1.** | Volumetric analysis | Burette pipette method | Double burette method | Generate as little waste as possible by using less quantity of solutions and increase safety for operator by avoiding mouth pipetting, keeping sample size and sample number |

| | | | | |
|---|---|---|---|---|
| | | | | minimum. [S. No. (3), (7), (10) (Table 2)] |
| **2.** | Inorganic Mixture Analysis | Group Analysis Method | Spot Analysis technique | Miniaturization of methods using spot plate, generating less waste, keeping sample size and sample number minimum. and eliminating toxic reagents like $H_2S$ gas. [S. No. (3) (5) (7), (10) (12) (Table 2)] |
| **3.** | Organic elemental analysis | Laissaigne's Method | Middleton method | Eliminating toxic reagents and increasing safety of the operator by replacing sodium metal with sodium carbonate and zinc dust mixture. [S. No. (7), (12) Table 2] |

After performing experiments at the end of academic session, a structured google form was circulated amongst the students which helped to explore students' experiences and their perception of green microscale techniques. Besides this, for a deeper insight a direct observational analysis by researcher and other faculty members was also carried out during laboratory sessions to find the efficient use of chemicals, safety of the students and adherence to microscale protocols.

**Procedure for Experiment 1: Volumetric estimation of magnesium ions in the given solution by complexometric titrations using disodium salt of Ethylenediamminetetraacetic acid (EDTA)**

Green analytical microscale technique: Double burette method.

This method carried out in chemistry laboratory by 52 selected students of undergraduate (UG) and post graduate (PG) classes for the estimation of magnesium ions in the test solution by complexometric titration using double burette method. The students selected were well aware of the conventional burette pipette method. The following steps were followed in the double burette method to get the end point.

1. Test solution of magnesium sulphate and Standardised solution disodium salt of EDTA (0.01M) were taken in two separate burettes marked 1 and 2 respectively and noted initial reading of burette 2 (V1)
2. 10 ml of magnesium sulphate solution was taken in the flask from burette 1 and it was diluted with 10 ml distilled water. 7 ml of ammoniacal buffer solution ($NH_4Cl + NH_4OH$) and 3 drops of freshly prepared Eriochrome black T (EBT) indicator were added to the same flask. A wine-red colour appeared. (Figure 1)
3. EDTA solution from burette 2 was added dropwise to the prepared flask with constant shaking till wine red colour changed to blue. This was the end point (Figure 2) Final reading of the burette 2 was noted V2 and amount of EDTA used calculated (V2-V1). One set of reading was obtained at this point.
4. Solution in flask was not discarded after the first set of readings unlike conventional method.
5. Repeated steps (6), (7) and each time noted the volume readings from the two burettes till five sets of readings were obtained.
6. The molarity of the magnesium sulphate solution was calculated for each set of burette reading using molarity equation at the end point. (wine red to blue) $M1V1\ (standard\ 0.01\ M\ EDTA\ solution) = M2V2\ (Magnesium\ sulphate\ Test\ solution)$

**Figure 1: Wine red colour (Magnesium sulphate solution in burette 1)**

**Figure 2: Blue colour (Disodium salt of EDTA solution in burette 2)**

**Procedure for Experiment 2: To analyse cations by spot test.**

Green Analytical microscale technique: Spot Analysis method

Spot tests which are simple and do not require costly equipment or apparatus and are of microscale nature, have been used in this experiment. "[11,12]"

The spot test was done on pilot basis by 52 selected students who had earlier performed the same experiments by the conventional group analysis method. The tests were performed for few basic radicals and students could easily deduce the radical by simple colour tests on a spot plate using one drop of the salt solution and one drop of the reagent. Placed one drop of test solution on the spot plate and added appropriate reagent. Noticed the colour of precipitate/ solution to confirm the ion.

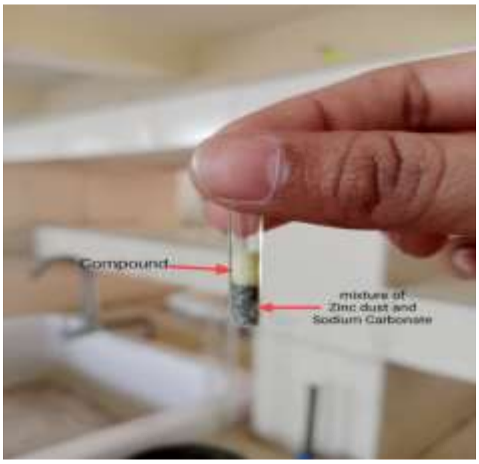
**Procedure for Experiment 3: Detection of extra elements in an unknown organic compound .**

Microanalysis technique 3: Middleton method for Elemental analysis of organic

The extra elements nitrogen, Sulphur and halogens in organic compound benzamide were analyzed by preparing the extract of the compound with sodium carbonate and zinc dust mixture instead of sodium metal used in the conventional Laissaigne extract method. A 1: 1 mixture of sodium carbonate and zinc dust was prepared (Figure 3) and a pinch of it added to the fusion tube followed by an equal amount of the organic compound. (Figure 4) The fusion tube was heated in the Bunsen burner flame till red hot and then plunged in a China dish containing distilled water. Similar process was repeated with another fusion tube. The contents of the China dish were boiled and filtered. The filtrate was used for carrying out tests for extra elements as per the conventional method only. "[13]"

**Figure 3. 1:1 Mixture of zinc dust and sodium carbonate in fusion tube**

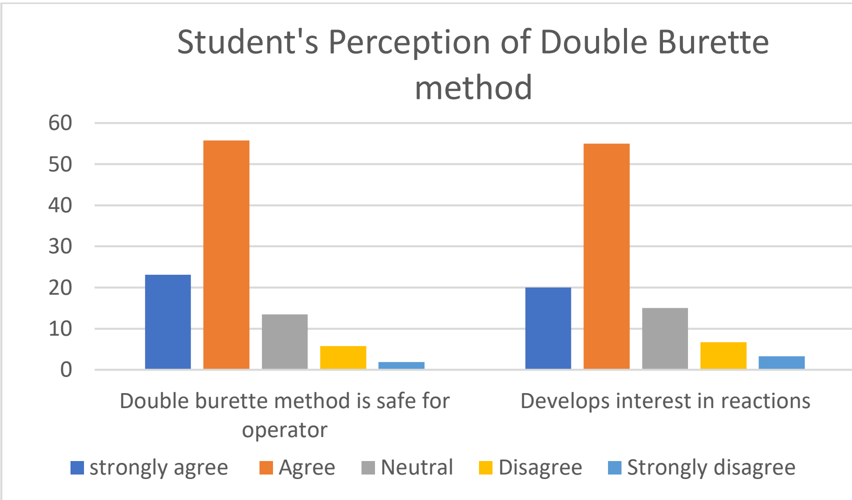

**Figure 4: Compound with 1:1 mixture of zinc dust and sodium carbonate**




**Results and Discussion:**

**Experiment 1:** Volumetric analysis by double burette method clearly followed the green principles as 8 ml of buffer solution and 4 drops indicator used as against 40 ml buffer and 20 ml indicator in conventional method. Magnesium sulphate solution used was 13 ml for five sets of readings as against 50 ml in conventional method 28 ml EDTA was used for five sets of readings as against 127 ml in conventional method. This resulted in less waste generation and developed greater interest amongst students by avoiding mouth pipetting. The structured google form data revealed that 55.8% of the students agree and 23.1% strongly agree with the fact that double burette method is safe for students as mouth pipetting is avoided in this method. 55% of the students agree and 20% strongly agree of that complexation reactions and colour change concept were clarified more easily by double burette method. (Figure 5)

**Figure 5: Student perception of Double Burette method**



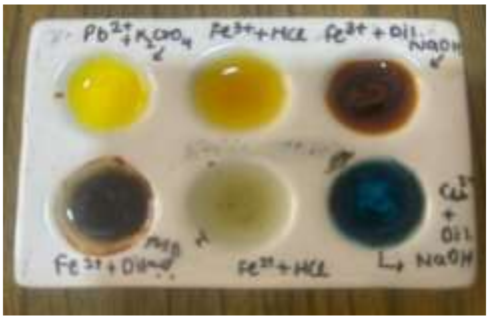

Observational analysis of the laboratory practical sessions revealed that the confidence level of students was heightened after performing the practical and they appreciated the less time consumed in performing practical. Similar observations have been made in few other researches. “[14]”

**Experiment 2**: Spot analysis method for identification of cations was a direct colour test (Table 4) with minimum miniaturised apparatus (Figure 6) and it clearly avoided the long cumbersome process of group analysis and generated less waste.

**Table 4: Spot tests for some cations**

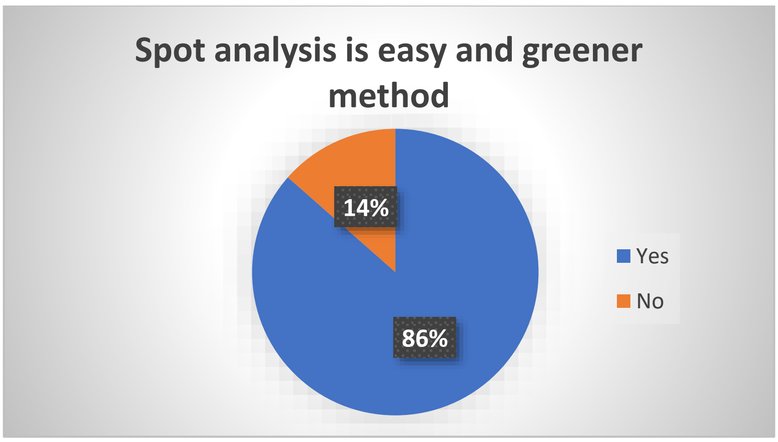
| S.N | Test solution | Reagent used | Colour Observed | Cation confirmed |
|---|---|---|---|---|
| | Ferrous sulphate | Potassium ferricyanide | Dark Blue(Turnbull's blue) | $Fe2+$ |
| | Ferric chloride | One drop Potassium thiocyanate and one drop hydrochloric acid (HCl) | Blood red colour | $Fe3+$ |
| | Ferric chloride | Sodium hydroxide | Reddish brown gelatinous precipitate | $Fe3+$ |
| | Copper sulphate | Sodium hydroxide | Light blue precipitate | $Cu2+$ |
| | Lead chloride | Potassium chromate | Yellow precipitate | $Pb2+$ |
| | Silver nitrate | Dilute Hydrochloric acid | White precipitate | $Ag+$ |

**Figure 6: Spot plate showing colour test for cations**



Survey through google form further confirmed that 86% of the students found the spot method greener, less complicated and student friendly as compared to the conventional group analysis method. (Figure 7)

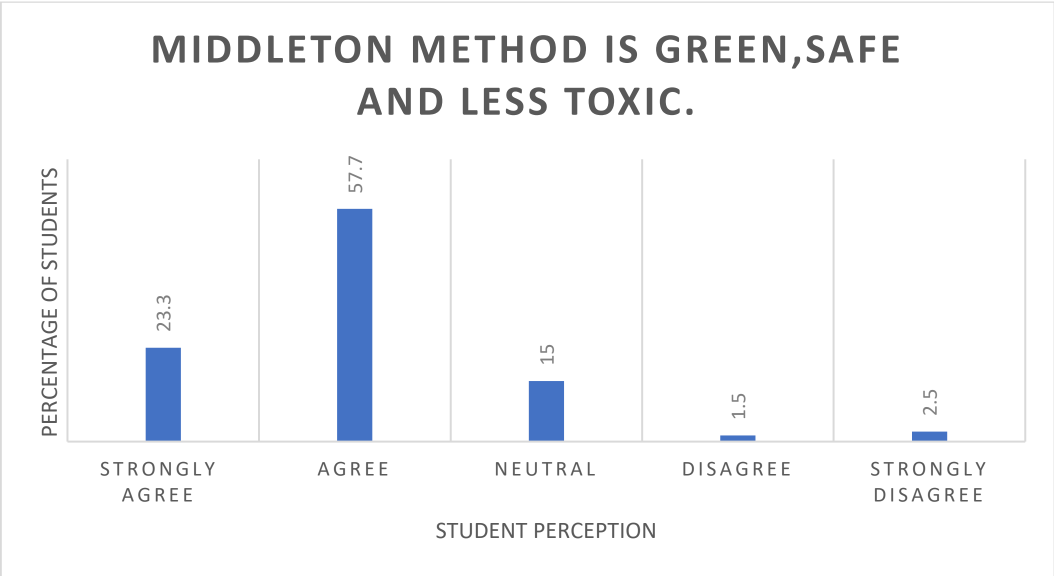
**Figure 7: Student perception of spot analysis method**



Observational analysis of laboratory practices showed that 75% of the students consider use of Hydrogen sulphide ($H_2S$) gas and repeated filtration to be the major cause of aversion amongst students for the conventional group analysis method.

**Experiment 3:** The elemental analysis of organic compound by middleton method followed the safety principle by using sodium carbonate and zinc dust. Google form survey showed that 23.3 % students strongly agree and 57.7% students agree on the statement that Middleton method is green, safe, less toxic and environment friendly. (Figure 8)

**Figure 8: Student perception of middleton method**

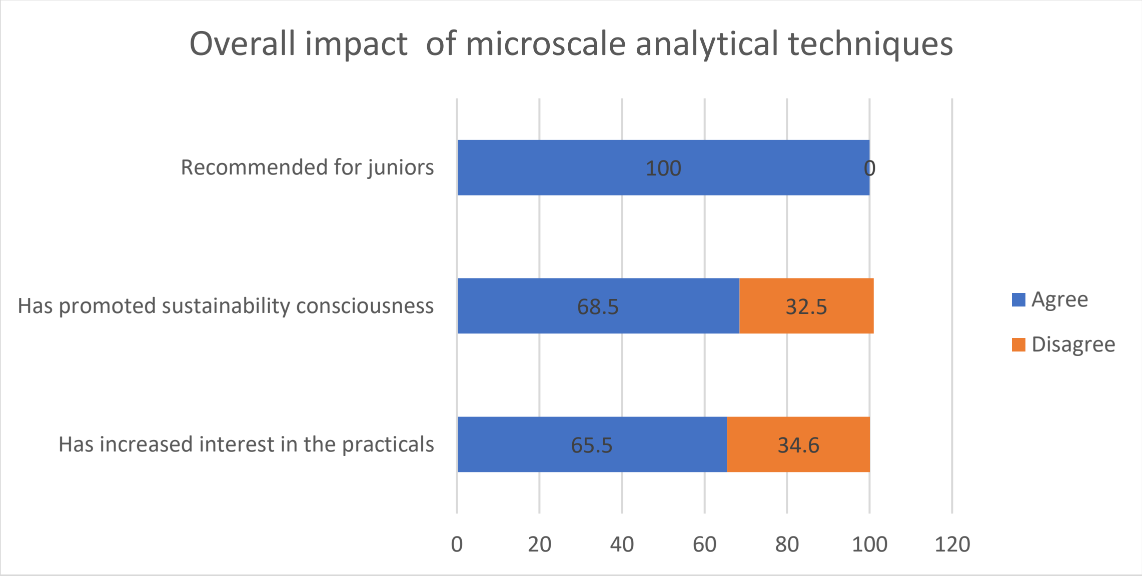



Observation of laboratory procedures showed a 60% increase in active participation of students in the analysis without fear of accidents due to sodium metal.

**Overall impact**:

68.5% students agreed with the fact that microscale techniques promote sustainability consciousness amongst the students. 65.5% students agree with a substantial increase in their interest in performing the chemistry practical after the adoption of these techniques and 100% of the students recommended them for their juniors and other bathcmates. (Figure 9)

Figure 9: Student perception on impact of microscale analytical techniques




**Conclusions:**
These findings indicate that if these microscale analytical techniques are incorporated in the UG and PG Practical curriculum, they can inculcate sustainable practices and can help in development of new green laboratories where reduced waste generation, safer handling of chemicals and simplified apparatus are the basic principles followed; thereby supporting goals of green chemistry. "[15,16]".

**Suggestions and future prospects:**
1. The present study opines that by incorporating microscale analytical technique students interest in the subject can be increased and this approach can be further extended to interdisciplinary domains like environmental science, pharmaceutical chemistry.
2. The green microscale techniques should be systematically inculcated in the higher secondary, UG and PG curriculum with revised laboratory manuals and innovative techniques for microscale experiments should be motivated.
3. The remaining seven revised principles of GAC can be studied in detail for their applicability to the various chemistry experiments done at school and college level laboratories.
4. Further research can explore how these green analytical micro techniques can impact the student's sustainability consciousness and positively influence the green chemistry and sustainability goals. "[15,16]"

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