



### RESEARCH ARTICLE

## RECOVERY AND REUSE OF NICKEL(II) FROM NI-DIMETHYL GLYOXIME COMPLEX: A SUSTAINABLE LABORATORY PRACTICE

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### Abstract

The current research study focusses on the recovery and reuse of Nickel chloride obtained from Ni-(DMG)<sub>2</sub> complex prepared during an undergraduate chemistry practical course. The complex was decomposed by using different acids followed by cooling at room temperature and refrigeration. Nickel (II) in the form of Nickel chloride, Nickel sulphate and Nickel nitrate was obtained after the acid digestion process. The recovered Ni(II) solution obtained was further reused for Ni (DMG)<sub>2</sub> complex preparation. The recovery and reuse technique are an attempt towards reducing the environmental footprint in an undergraduate chemistry laboratory and towards sustainable laboratory management.

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### Introduction:-

Sustainability is increasingly becoming the guiding principle for contemporary company strategies, and laboratories are no different. Nevertheless, operating a laboratory consumes a substantial quantity of energy and materials, resulting in increased expenses for both the financial aspect and the environment. As a reaction to this, numerous laboratories and pharmaceutical businesses are implementing measures to ensure the long-term viability of their operations [1]. A sustainable laboratory employs strategies to minimize its ecological footprint. Some notable characteristics of this specific ecosystem include minimizing waste, improving systems to optimize efficiency, implementing recycling and waste management protocols [2].

Laboratory courses are a ubiquitous method of instruction in the field of chemistry. In a paper entitled "The Advantages of Laboratory Work in the Study of Elementary Chemistry," Bowers argued that laboratory work is

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necessary to develop observational skills [3]. In his study he devised an assessment consisting of a total of 20 inquiries, with half of them focusing solely on material covered in the textbook and the other half encompassing topic taught in both the textbook and laboratory. He administered this exam to students who had finished their first-year university laboratory and assessed the precision of students' answers depending on the duration since they had completed their laboratory work at the university. Students demonstrated higher performance on material covered in both the textbook and laboratory, as opposed to material covered only in the textbook. Laboratory experiments yield greater learning outcomes compared to relying solely on the textbook. The American Chemical Society Committee on Professional Training (CPT) mandates that bachelor's degrees in chemistry must include a minimum of 400 hours of additional laboratory work, in addition to the basic chemistry courses taken in the first year of university [4]. According to the CPT, laboratory experiences offer a highly appealing chance for conducting investigations that are led by inquiry and open-ended. These experiences foster autonomous thinking, critical thinking, reasoning, and a perspective of chemistry as a scientific process of exploration [5].

The study of coordination compounds is a significant and demanding field within contemporary inorganic chemistry. Recent advancements in the understanding of chemical bonding and molecular structure have yielded valuable knowledge on the role of these compounds as essential elements in biological systems. Chlorophyll, hemoglobin, and vitamin B12 are complex molecules that contain magnesium, iron, and cobalt, respectively [6]. Coordination compounds are utilized in a range of metallurgical processes, industrial catalysts, and analytical reagents [7]. Coordination compounds are widely utilized in electroplating, textile dyeing, and pharmaceutical chemistry. Coordination chemistry is an important component of the syllabus of undergraduate chemistry in University of Mumbai. The syllabus incorporates the various theoretical aspects of coordination chemistry. The practical component of coordination chemistry focusses on the synthesis of coordination compounds. One of the various experiments included in the syllabus is synthesis of Nickel -Dimethylglyoxime complex

The preparation of Ni (DMG)<sub>2</sub> complex has been incorporated in the undergraduate chemistry syllabus of University of Mumbai by way of three experiments

1. Gravimetric estimation of Nickel as Nickel Dimethylglyoxime
2. Inorganic preparation of Nickel Dimethylglyoxime complex
3. Green Synthesis of Nickel Dimethylglyoxime complex

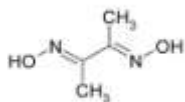
The performance of these practicals in our undergraduate chemistry lab resulted in the preparation of 92 g of Ni-(DMG)<sub>2</sub> complex in the academic year 2023-24. The disposal of the synthesized Ni-(DMG)<sub>2</sub> complex poses a challenge. In order to keep up with the goal of sustainable laboratory practice a study was planned to study various methods for the recovery and reuse of NiCl<sub>2</sub> from Ni-(DMG)<sub>2</sub> complex. The study was carried out in such a way that the product generated in an experiment could be reused by a next set of students by incorporating a recovery step in the experimental procedure.

### Material and Methods:-

The Ni (DMG)<sub>2</sub> complex prepared during regular practical course of the academic year was stored in labeled, air tight containers. 1 g of Ni (DMG)<sub>2</sub> complex was decomposed using 10 cm<sup>3</sup> of concentrated acid. The dissolution was performed in three batches by using Conc HCl, Conc HNO<sub>3</sub> and Conc H<sub>2</sub>SO<sub>4</sub>. The solution obtained after leaching the complex with acids was subjected to cooling at room temperature and under refrigeration. The solution obtained was diluted to 100 cm<sup>3</sup>. 50 cm<sup>3</sup> solutions obtained were then reused for the synthesis of Ni (DMG)<sub>2</sub> complex. The complex was prepared by the addition of 1% DMG solution to recovered Ni (II) solution in an ammoniacal medium [8].

#### Reactants:

- 1) Nickel
  - Symbol: Ni
  - Atomic Number = 28
- 2) Dimethylglyoxime
  - Molecular Formula: C<sub>4</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>



- 1% reagent solution is prepared by dissolving 1g of DMG in 100cm<sup>3</sup> of ethyl alcohol
- DMG is a bidentate ligand which gets attached to the metal ion at two points.
- The complex formed is a chelate complex

### Reaction

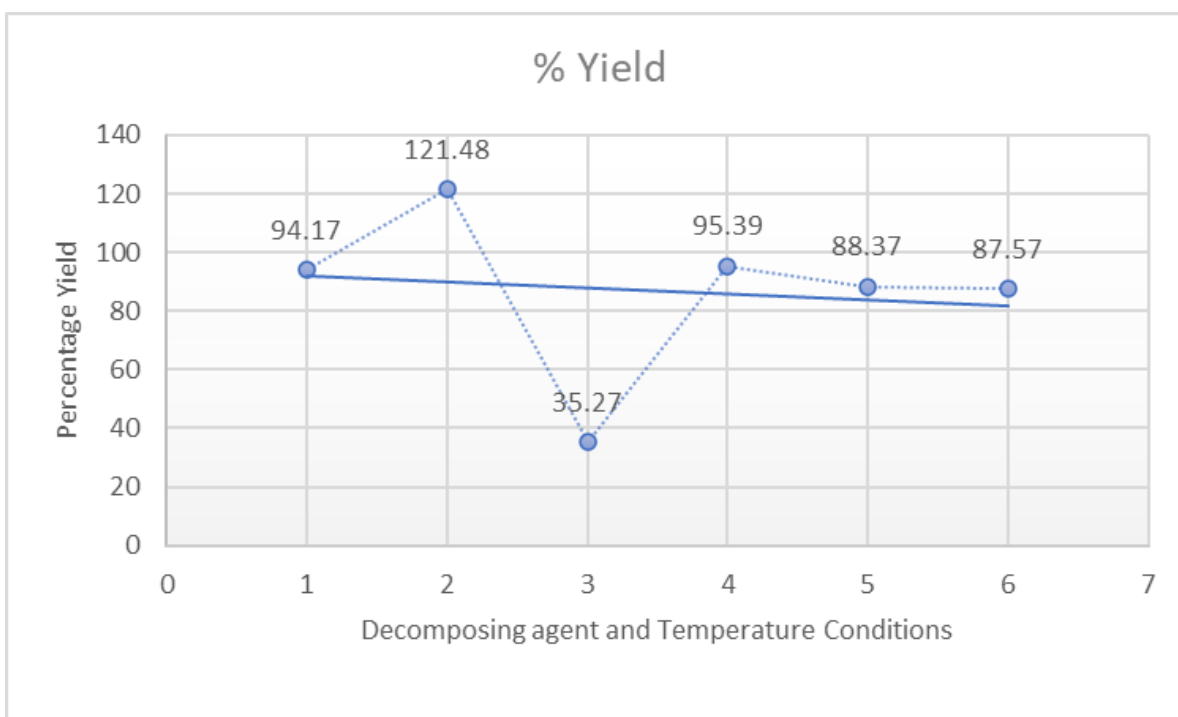
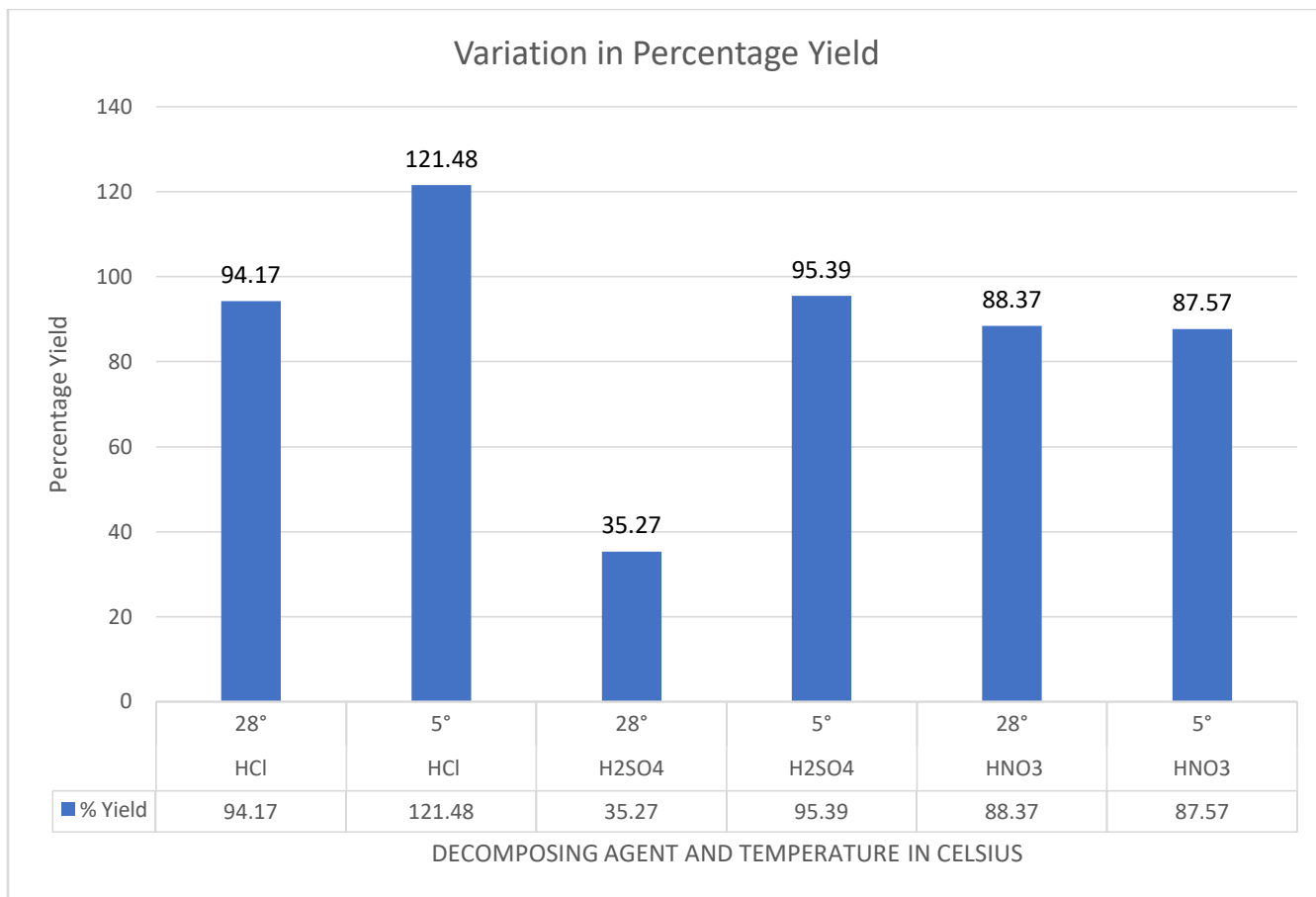
### Results and Discussion:-

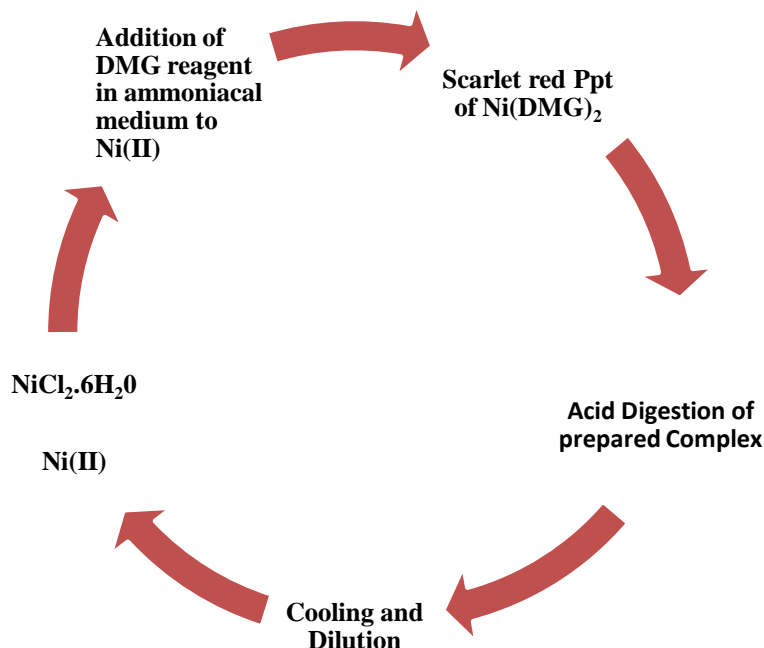
The addition of 1% DMG reagent to the ammoniacal solution of recovered Ni (II) solution resulted in the formation of scarlet red colored Nickel Dimethyl glyoxime complex in all three sets of experiment. The yield of the complex obtained from the recovered solution using three different acids are as follows:

Decomposing agent	Temperature	Observed Yield of Ni (DMG) <sub>2</sub> in g	Theoretical Yield of Ni (DMG) <sub>2</sub> in g	% Yield
HCl	28 <sup>0</sup> C	0.469	0.498	94.17
HCl	5 <sup>0</sup> C	0.605		121.48
H <sub>2</sub> SO <sub>4</sub>	28 <sup>0</sup> C	0.176	0.499	35.27
H <sub>2</sub> SO <sub>4</sub>	5 <sup>0</sup> C	0.476		95.39
HNO <sub>3</sub>	28 <sup>0</sup> C	0.441	0.499	88.37
HNO <sub>3</sub>	5 <sup>0</sup> C	0.437		87.57

The yield of Ni (DMG)<sub>2</sub> obtained from the recovered solutions were in the range of 35.27% to 121%. The results obtained for the solutions recovered using Conc HCl followed by cooling at 5<sup>0</sup>C and Conc H<sub>2</sub>SO<sub>4</sub> followed by cooling at room temperature were the outliers. However, a linear trend line could be observed with the percentage varying between 87% to 95%

During a regular practical usually a 5% solution of Nickel chloride is prepared for the synthesis of Ni (DMG)<sub>2</sub>. 25cm<sup>3</sup> of the stock solution is taken by the learner to carry out the synthesis. This translates to 1.250 g of NiCl<sub>2</sub> in the stock solution. In a batch of 20 students 25 g of NiCl<sub>2</sub>.6H<sub>2</sub>O is used for the preparation. And in a class of 60 students 75 g of NiCl<sub>2</sub>.6H<sub>2</sub>O is used for the synthesis of Ni (DMG)<sub>2</sub> complex. The recovery method using Conc HCl at room temperature and Conc H<sub>2</sub>SO<sub>4</sub> at 5<sup>0</sup>C gave a percentage yield of 94.17% and 95.39% respectively. One of these two methods can be incorporated as the last step in the experimental procedure to recover Ni (II) solution. The recovered Ni (II) solution thus can be used by the next set of students to perform the experiment. This step will eliminate the use of fresh NiCl<sub>2</sub>.6H<sub>2</sub>O and thus create a circular economy management protocol in the laboratory.





### Circular Economy Model In The Chemistry Laboratory

The above study demonstrates that step of decomposition of the synthesized Ni (DMG)<sub>2</sub> complex can be included as the last step the experiment so that the recovered Nickel Chloride solution can be used for the synthesis of Ni (DMG)<sub>2</sub> by the next batch of learners. The experiment thus becomes a closed loop experiment where the product of one experiment can become the starting material for the next set of learners. There are several instances of closed loop experiment available in literature. The College of the Redwoods, situated in California, has developed a sequence of "closed loop" experiments in which the residual substances from one experiment are utilized as the reagents or reactants in the subsequent experiment [9]. Upon the conclusion of the series, the residual outcomes of the ultimate experiment are made accessible for the subsequent cohort of students to initiate the cycle anew. Aqueous laboratory nano waste is used to extract gold and convert it into an aqueous solution of H<sub>2</sub>AuCl<sub>4</sub>. This is achieved using straightforward methods employing easily accessible chemical reagents such as NaCl, HCl, and H<sub>2</sub>O<sub>2</sub>. The H<sub>2</sub>AuCl<sub>4</sub> solution obtained is subsequently utilized to generate spherical Au nanoparticles via a seed-mediated method[10]. Chemical recovery can be a helpful educational tool for students and can be given as the concluding stage of a chemistry experiment or as an optional opportunity for interested students to earn extra credit. Recovery treatments must always be conducted under supervision.

### Conclusion:-

Chemicals are indispensable to our daily existence. However, the usage and production of chemicals also have a substantial impact on the environment and contribute to climate change. Chemical innovation plays a crucial role in tackling sustainability and climate change challenges. To mitigate the adverse impact of chemicals on the environment while simultaneously meeting social needs, it is imperative to pursue chemical innovation that promotes sustainability. The conventional linear approach of 'take-make-dispose' is being replaced by a circular economy mindset in the chemical sector. There is a growing emphasis on developing closed-loop systems that view waste as a useful resource. The ongoing research we are conducting involves the recycling and reuse of Ni (DMG)<sub>2</sub> that was synthesized in an undergraduate laboratory. This work aims to promote a circular economy model and contribute to a sustainable future. To enhance chemistry instruction at all levels, it is imperative to include multiple closed loop experiments in the existing curriculum design. This, in turn, will decrease the ecological footprint of chemistry laboratories.

### Abbreviations:

Ni: Nickel

DMG: Dimethyl Glyoxime

**Conflict of Interest:**

None.

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