ELECTRONIC SUPPLEMENTAL INFORMATION. Leveraging large language models (LLMs) to enhance student inquiry in a nanochemistry teaching laboratory: a Fenton-like oxidation using recyclable ferrite nanoparticle (NP) catalysts.

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S1 Nanochemistry course offerings in Canadian and United States universities

We note at the outset that this is not meant to be a comprehensive list. The course offerings are based on information obtained from university course catalogues found online. We regret any omissions or outdated information. We are also aware that many educational institutions in Europe, Australia, Asia, and the global South in general offer nanochemistry courses, but a global survey is beyond the scope of this section.

S1.1 Selected examples of institutions in USA and Canada offering dedicated UG nanochemistry teaching laboratories or lecture/lab combined courses

1. University of Southern California - CHEM 463L *Upper level undergraduate lab*
2. University of Puget Sound - CHEM 324 *Upper level undergraduate lecture + lab*
3. Rice University - CHEM 367 *Upper level undergraduate lab*
4. Beloit College - CHEM 150 *rotating lecture + lab offering*
5. Carleton University - CHEM 3107 *experiential learning activity*
6. University of Calgary - NANS 502 *Upper level undergraduate lab; co-coded with chemistry*
7. **Colorado State University - CHEM 312** *subject of this report*

S1.2 Surveying UG nanochemistry offerings at select North American institutions

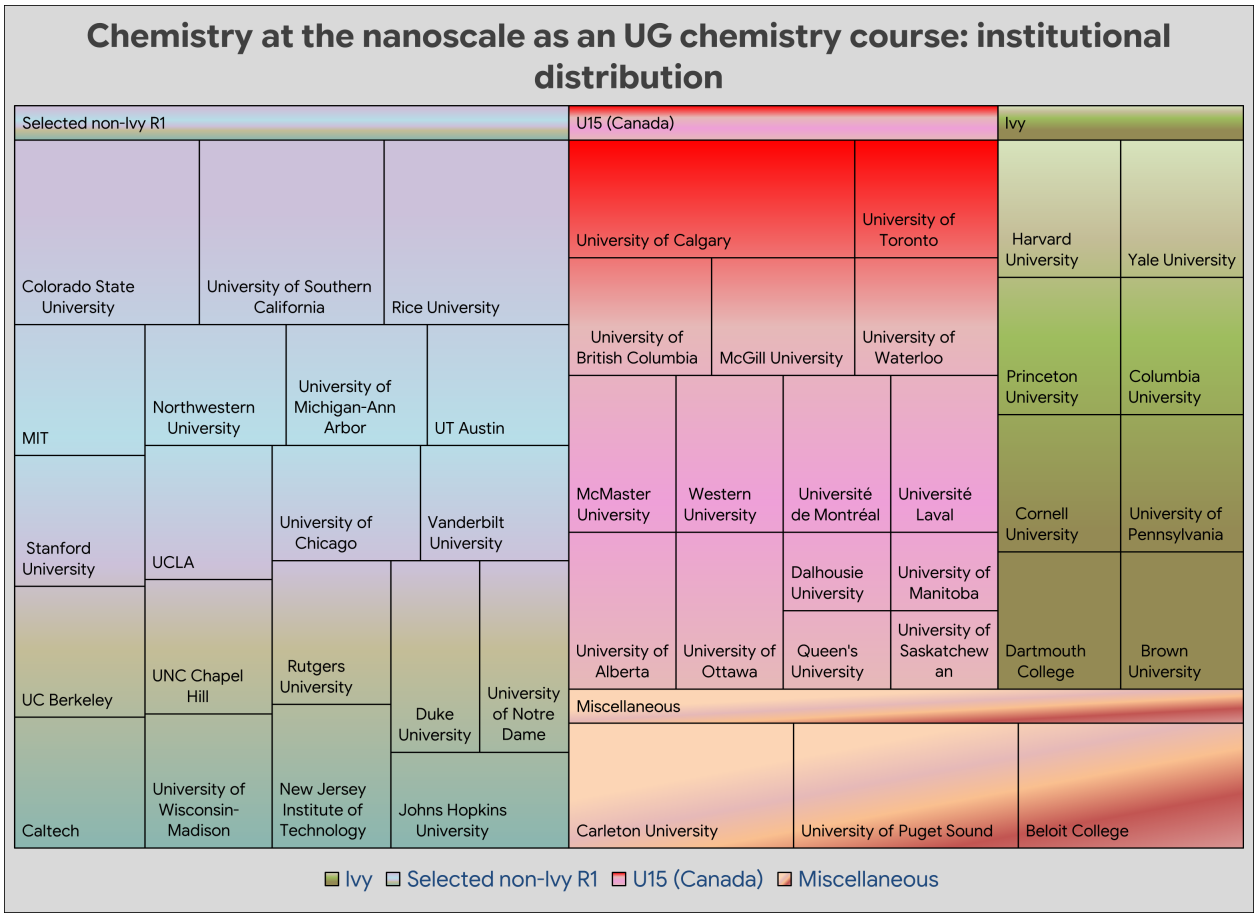

Fig. S1 A tree-map of universities in Canada and US offering nanoscale chemistry courses in their UG curriculum. A large box corresponds to a score of 2, implying the existence of nanochemistry lecture and laboratory courses; a medium box corresponds to a score of 1, indicating (mostly) nanochemistry lecture courses without explicit mention of a corresponding laboratory course; and a small box represents a score of 0.5, indicating topics related to nanochemistry being covered as part of a more general course such as chemistry of materials, colloids and interfaces, and the like.

Definitions:

1. The Ivy League is an athletic conference and informal label for eight private research universities in the northeastern United States.
2. R1 (Doctoral Universities; very high research activity) is a Carnegie Classification category for U.S. universities that award a large number of doctoral degrees and conduct a very high level of research, measured by things like research expenditures and number of research-doctorate graduates.
3. U15 (also written U15 Group of Canadian Research Universities) is a consortium of 15 large, research-intensive universities in Canada, classified by metrics such as research funding, PhD output, and research infrastructure.

n.b.- We only surveyed B.S./B.Sc. Chemistry course catalogues, including co-coded courses; specialized nanoscience degrees, certifications, and nanoscience courses offered by other departments such as engineering or biology were not taken into account.

S2 Plan of work

1. **Day 1 -** Synthesis of MMFNPs; separation.
2. **Day 2 -** UV-Vis; IR; DLS; sample preparation for SEM and PXRD.
3. **Day 3 -** Visit to ARC microscopy lab for SEM; FiJi tutorial; data processing.
4. **Day 4 -** PXRD data processing tutorial, introduction to Mercury and other PXRD software; lecture on SEM data processing using FiJi.
5. **Day 5 -** Catalytic application of MFe_2O_4/tween-20 in the removal of methylene blue from simulated wastewater.

S3 Sample pre-lab questions

1. Select ALL the classes of hazardous material(s) that you will be handling during the synthesis of the MMFNPs: **strong acids, strong bases, corrosives**, explosives, lachrymators.
2. True or false: if you are already wearing prescription glasses, you no longer need safety glasses. **False!**
3. If Google Gemini provides a DOI of a scientific publication, do you: (a) accept it as-is; (b) click on the link to make sure that the publication exists; or **(c) click on the link, access the publication, and read the relevant section to verify the information summarized by the LLM**?

S4 Extract from student manual: instructions for MMFNP synthesis

Miscellaneous Instructions

1. Please use appropriate PPE for this laboratory - gloves, closed toed-footwear, lab coats and eye protection at all times.
2. You will deal with chemicals such as metal chlorides dissolved in strong acid and NaOH solution. They are toxic and/or corrosive. NP slurries are very difficult to remove from fabric and other surfaces. Contact with skin and clothing should be minimized.
3. Specific hazards: Hydrochloric acid is corrosive, and the base is also corrosive (though dilute in this lab). We will be combining acids and bases in this experiment; the reaction should be carried out slowly to prevent overheating of glassware. Metal chlorides can be corrosive, toxic and/or mutagens.
4. Consult the MSDSs of the materials you will be working with; your pre-lab quiz will test you on this.
5. Follow the TA instructions on safe lab conduct, waste disposal as per EHS regulations, and proper lab techniques.
6. Training in the use of UV-Vis, DLS, and ATR-IR will be provided. You are responsible for your own measurements, data collection, and retrieval. Help with the interpretation will be provided.

Instructions for the synthesis of Tween-20 capped ferrite NPs.

1. You are synthesizing spinel NPs with the general formula MFe_2O_4 ($M^{2+} = Mn^{2+}, Ni^{2+}, Co^{2+}$) by co-precipitation. You will do the reaction in a 3-necked round bottom flask; this shall be provided to you. Make sure it is clean.
2. Prepare individually the precursor solutions of the divalent metal salt (10 ml, 0.25 M) and iron (III) chloride hexahydrate (10 ml, 0.5 M) and heat them to *ca.* 60°C.
3. Prepare a gently boiling solution of NaOH (120 mL, 0.7 M) in DI water under a nitrogen gas flow.
4. To the NaOH solution add enough Tween-20 (molar mass is 1227.54 $g \cdot mol^{-1}$) to prepare a *ca.* 2 mM solution.
5. While continuing the nitrogen flow, inject *simultaneously* both the metal precursor solutions to the NaOH solution containing the Tween-20. The TA will help you with this step.
6. Set up a reflux condenser and heat your reaction mixture to reflux for 60 min. You may stop the nitrogen flow after 30 min, but do not expose your system to air. The TA will demonstrate the assembling of a reflux setup beforehand.
7. After 1 h, stop the reflux, and cool to room temperature.
8. Recover the MFe_2O_4 NPs through centrifugation and wash twice with 2 mM Tween-20 solution. Finally, label this as *YourInitials-312-[metal] ferriteNP* and leave it to dry in the centrifuge tube.
9. Follow the TA's instructions for the clean up.

S5 Extract from student manual: catalytic decomposition of methylene blue dye

1. This part of the experiment will be performed in groups of two. The TA will allocate the groups.
2. Set up the UV lamp but DO NOT turn it on yet - H_2O_2 and the dye are photosensitive.
3. Add 5 mL of 30% H_2O_2 from the refrigerator to 95 mL of the supplied 16 μM methylene blue solution in a 200 mL beaker.
4. To this add the supplied 2 M NaOH solution dropwise to raise the pH of the system approximately to 11. Check the pH using a pH-meter.
5. Meanwhile, familiarize yourself with the spectrophotometer using the instructions taped to the bench. Obtain a cuvette, fill it with DI water, and measure the baseline.
6. Take a 1 mL aliquot of the solution and dilute with 1 mL DI water. Obtain the 't=0' spectrum of the dye solution prior to addition of the MMFNPs.
7. Turn on the UV lamp and add 25 mg of the dry MMFNPs. Stir vigorously for 30 seconds with a glass stir rod. Start the timer.
8. Note any changes in the colour of the dye solution after this step.
9. After agitating with the glass rod, set magnetic stirring to 100 rpm and allow the beaker to sit uninterrupted as the reaction progresses.
10. Withdraw a 1 mL aliquot from the reaction beaker every 10 minutes to record the UV-visible spectra. Remember to dilute properly and wash the cuvette between runs with DI water. Record the exact time of each run.
11. After taking your '60 minute' aliquot, we will consider the reaction 'finished' for the purposes of this lab. Cease stirring, allow the solution to settle and the MMFNPs to aggregate on the stir bar.
12. Save and export spectral data from your experiment.
13. We will repeat the experiment twice more with THE SAME MMFNPs to illustrate the recyclability of the NPs. Prepare a fresh beaker of dye solution as instructed in Steps 3-6. To begin the next reaction, transfer the NP-covered stir bar from the previous reaction to the new solution. Proceed as instructed in Steps 7-11.
14. Clean the cuvette, collect your spectral data on your USB drive following the instructions issued by the TA, and clean up after yourself.

S6 Tauc plot from UV-Vis data

In the X-axis, plot energy.

λ is the wavelength column of your UV-Vis data. Inserting the numerical values of the speed of light (c) and Planck's constant (h), we obtain:

$$E = h \cdot \nu = \frac{h \cdot c}{\lambda} = \frac{1240\,(\mathrm{eV} \cdot \mathrm{nm})}{\lambda\,(\mathrm{nm})} \tag{S1}$$

In the Y-axis, you will plot $(\alpha \cdot h \cdot \nu)^n$, where $h \cdot \nu$ is the incident photonic energy and α is the absorbance coefficient of the MMFNPs, which can be obtained from the Y-axis data of the UV-Vis plot (A).

$$\alpha = ln(10) \cdot A \tag{S2}$$

$$Y = [ln(10) \cdot A \cdot E]^2 \tag{S3}$$

Note: n equals 2 here, for direct band gap estimation.

Now, plot Y as a function of E and extrapolate the rising edge of the curve to $Y = 0$. The corresponding E, denoted as E_g, can be read directly from the X-axis, and is the direct band gap for your MMFNPs.

S7 PXRD data

Immediately after collection, the data were processed by the ARC's crystallographer (EC) using *Diffrac.EVA V7.1.0.2*. The data were imported into *Diffrac.EVA* to assess sample purity, phase identification, and exported the data as *.xy* files so students could process the data and perform phase identification without the use of proprietary software. Additionally, a second data file was created with the Cu $K_{\alpha 2}$ wavelength signals removed from the data using *Diffrac.EVA*'s built in stripping tool. Peak position and full width at half maximum (*fwhm*) for each observable diffraction peak were calculated by using *Diffrac.EVA*'s Create Area tool. Note that this tool also outputs average crystallite size using the Scherrer equation (see below) but these values were ignored and calculated separately in a method the students could reproduce without the software. Different diffractometers will utilize different software packages for analysis of data but all should be able to output a basic text file containing the diffraction data that can be analyzed without the need for instrument specific software.

The peak position and *fwhm* were converted to radians and used to approximate the average crystallite size using the Scherrer equation:

$$\tau = \frac{K\lambda}{\beta cos\theta} \tag{S4}$$

where τ is the average crystallite size, K is dimensionless shape factor with value depending on crystallite shape (we use a value of 0.89 for spherical crystallites), λ is the X-ray wavelength used in the experiment, β is the *fwhm* of the peak in radians, and θ is the peak position in radians.[82] Rearranging equation (2) to

$$\beta = \left(\frac{K\lambda}{\tau}\right)\frac{1}{cos\theta} \tag{S5}$$

we obtain a linear equation with slope related to the average crystallite size and a y-intercept of 0. The data is plotted as *fwhm vs.* cos θ and a linear fit is performed with y-intercept set to 0 to obtain the slope. The value for τ can then be obtained by:

$$\tau = \frac{K\lambda}{m} \tag{S6}$$

where m is the slope obtained from the linear fit. The value of τ is used to approximate the average crystallite size for the samples and is reported in the results section. A sample Scherrer plot for $CoFe_2O_4$ NPs is presented in Fig. **??**.

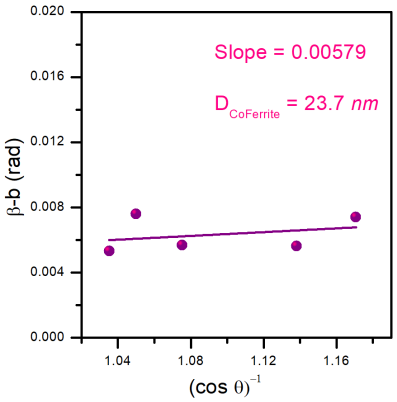

Fig. S2 Scherrer plot for the crystallite size determination of $CoFe_2O_4$ NPs.

As we can see from Fig. S3, $NiFe_2O_4$, in contrast to the other two ferrites, appears to be amorphous. Nickel ferrite often needs higher thermal energy or longer aging to crystallize than manganese and cobalt ferrites, so the same coprecipitation recipe produces amorphous $NiFe_2O_4$ while $MnFe_2O_4$ and $CoFe_2O_4$ show clear spinel peaks in PXRD. Differences in cation chemistry, cation distribution, and resulting nucleation/growth rates make $NiFe_2O_4$ more prone to stay as very small or poorly ordered nuclei under mild conditions.

S8 DLS size distribution data

The DLS size distribution data for all three nano-ferrites show a bimodal size distribution profile, with a smaller peak in the tens of nm size domain, and a larger one around 150-200 nm, likely owing to aggregation of the individual NPs or post-synthetic growth.

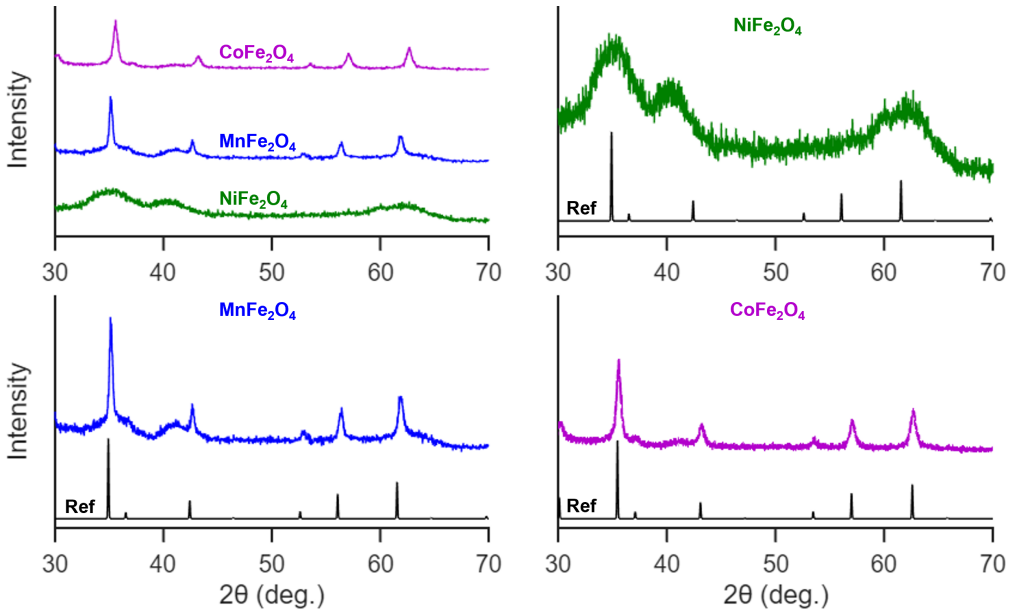

Fig. S3 (top left) composite figure showing PXRD of the as-synthesized MMFNPs; other panels superimpose the individual diffractograms on match-stick plots of the standard spinel phases from literature.

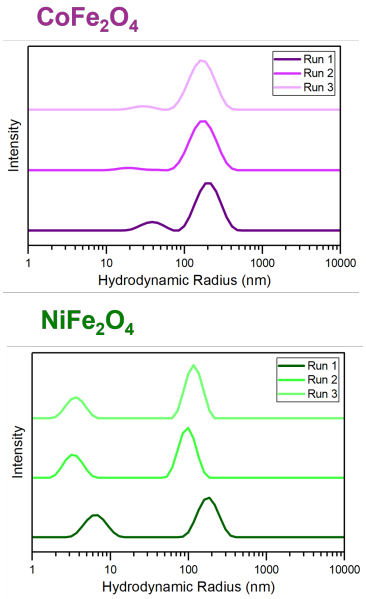

Fig. S4 Size distribution profiles for Tween-20 capped $CoFe_2O_4$ and $NiFe_2O_4$ obtained from DLS

S9 Integration of ethical LLM usage within the CURE

S9.1 Sample LLM prompts

To ensure that the integration of LLMs fosters genuine inquiry and critical thinking, our pedagogical design contains some basic concepts of prompt engineering and socratic interaction. Some examples are provided underneath:

1. **Pre-Synthesis: Predictive Reasoning**
   Students are expected to use a comparative prompt that requires them to justify their reasoning.

   Structured Prompt: "I am synthesizing MFe_2O_4 nanoparticles where M is Mn, Co, or Ni using co-precipitation. Based on the crystal field stabilization energy (CFSE) and ionic radii of these metals, predict which ferrite will likely exhibit the highest catalytic activity for methylene blue degradation. Provide a rationale for your prediction that I can later test against my experimental data."

Pedagogical Goal: This forces students to connect theoretical inorganic chemistry concepts to the experimental outcomes they are about to observe.

2. **Characterization: The "Reviewer" Role**
   Rather than asking the LLM to identify a peak, students can treat the LLM as a peer reviewer or an always-available lab supervisor.

   Structured Prompt: "I have obtained an XRD pattern for my synthesized $CoFe_2O_4$ NPs. The primary peak is at $2 \cdot \theta = 35.4^o$. The Scherrer equation gives me an average crystallite size of 12 nm. Check my calculation for errors and suggest two potential reasons why my experimental peak might be broader than the theoretical standard."

   Pedagogical Goal: This encourages students to double-check their work and consider physical factors like lattice strain or instrumental broadening.

3. **Green Chemistry Optimization**
   To address SDG 6 (Clean Water), students can use the LLM to move beyond the provided protocol.

   Structured Prompt: "The current lab protocol uses methylene blue as a model pollutant. Identify three limitations of using methylene blue to represent industrial textile wastewater. Suggest a 'green' modification to our current Fenton-like catalyst system that could improve the degradation rate of more complex organic dyes without increasing toxicity.".

   Pedagogical Goal: This aligns with the "Authentic Research" goal by asking students to think about the scalability and limitations of their "mini-project."

S9.2 LLM usage verification log and transparency

To prevent over-reliance, we insist that students maintain a simple verification log related to their LLM usage, to be shown every week to the GTA as a 'prelab' exercise. For every LLM output, students must:

1. Highlight any technical claims made by the LLM.
2. Verify those claims using a peer-reviewed source or their own experimental data.
3. Reflect on whether the LLM's initial hypothesis (from the pre-synthesis stage) was supported by their results.

This approach ensures that students are repeatedly exposed to the idea that LLM is not a 'magic box' producing answers out of thin air, but a tool that requires professional skepticism and scientific validation—skills that are essential for any student entering a modern, AI-integrated research environment. A model validation and verification log is shown in Table S5.

S10 Questions for LLM-guided active inquiry

This is an area where students are encouraged to use Google Gemini to find out not only the answers to these questions, but also the sources for those answers, and record everything in their LLM verification log. These questions can be included in the post-lab assignments. They also serve as points to address in the final student report and assist in grading, in the sense that students who address these in their final reports get higher grades.

1. **DLS:** What is the average ζ potential value of the suspension of MFe_2O_4 NPs in water? What can you conclude about the stability of the aqueous dispersion of the MMFNPs based on the numerical value of the ζ potential?
2. **SEM:** Why is the average diameter obtained from the SEM micrographs different from the one obtained from DLS?
3. **PXRD:** Why is $NiFe_2O_4$ amorphous while the other ferrites are crystalline? What implications would this have if we were to compare the catalytic efficiencies of the three ferrites for the degradation of methylene blue in water?
4. **catalysis:** From your study of the relevant literature, come up with a reasonable mechanism for the catalytic degradation of methylene blue that you performed. Can you think of an alternate source of UV-rich light which may be used instead of a UV lamp to promote the catalytic degradation?

Mechanism: The reaction mechanism associated with Fenton-type reactions, as proposed by Haber and Weiss, can explain the ferrite-catalyzed production of hydroxyl radicals for methylene blue degradation. Presence of a second $3d$ transition metal such as cobalt within the iron oxide lattice can also activate hydrogen peroxide for increased production of hydroxyl radicals, thus increasing

Segment	LLM Prompt	AI Claim/Output	Validation Source	Result/Reflection
Synthesis	Why do we obtain amorphous $NiFe_2O_4$ NPs, while the other ferrites show crystallinity? Is there a way to increase the crystallinity of $NiFe_2O_4$ NPs?	Nickel ferrite often needs higher thermal energy or longer aging to crystallize than manganese and cobalt ferrites; heating in a furnace at high temperatures (>450°C) can help increase crystallinity.	Cherpin *et al.*, *Materials*, **2021**, *14*(10), 2557. https://doi.org/10.3390/ma14102557	**Verified:** Crucial for understanding the diffractogram of $NiFe_2O_4$ NPs and catalytic performance comparison.
PXRD	Which is a common PXRD peak for all these ferrites?	The peak at 2θ=35.4°; Miller index **(311)**	COD Database / Mercury™	**Verified:** Matched experimental diffractogram peak at ~35°.
Application	Suggest two ways in which the MMFNP catalyzed Fenton-like oxidation of dyes in wastewater may be implemented at scale in a real-life scenario.	An industrial water treatment plant would use a reactor exposed to sunlight when possible, with back-up industrial UV-LEDs. The reaction would be done under continuous flow conditions.	This paper demonstrates a continuous 65-day operation in a secondary effluent plant, calculating a cost of **USD per ton** of water. Wang *et al.*, *Appl. Sci.* **2025**, *15*, 8210. https://doi.org/10.3390/app15158210	**Inconclusive:** No way to implement these recommendations within a teaching laboratory setup.

Fig. S5 Sample LLM verification and validation log.

the efficiency of the catalyst. Co^{3+} can be recycled to Co^{2+} by Fe^{2+} due to the thermodynamically favorable redox reaction, thus accelerating the decomposition of hydrogen peroxide. Apart from the radical mediated production of oxygen gas mentioned above, surface oxygen vacancies present in metal oxides can also decompose hydrogen peroxide directly into oxygen and water without producing intermediate hydroxyl radicals. Any or all of these mechanistic steps may be operating in tandem within our system.

- $Co^{2+}_{surface} + H_2O_2 + e^- \longrightarrow Co^{3+}_{surface} + OH^- + {}^{\cdot}OH$
- $Co^{3+}_{surface} + Fe^{2+}_{surface} \longrightarrow Co^{2+}_{surface} + Fe^{3+}_{surface}$
- ${}^{\cdot}OH/{}^{\cdot}OOH + \text{dye} \longrightarrow H_2O +$ colorless oxidized dye fragments.

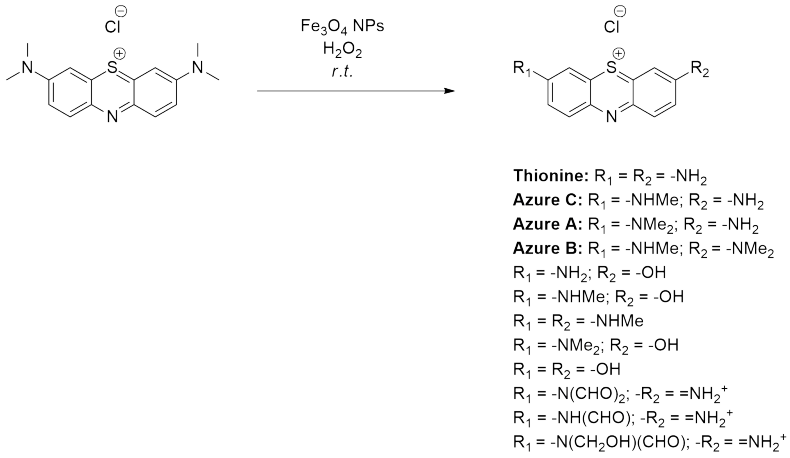

Fig. S6 Methylene blue decomposition products from mass spectrometry; data from Zubrik *et al., Sci. Rep.*, 2023, 14773.

S11 Sustainability spotlight

The CURE described here advances UN Sustainable Development Goals 4, 6, and 10 by blending digital literacy with green nanocatalysis. By integrating LLM-assisted optimization of the synthesis of nano-MFe_2O_4 ($M = Mn^{2+}, Co^{2+}, Ni^{2+}$) NPs and catalytic dye degradation into undergraduate labs, the project modernizes curricula with AI-driven modules (SDG 4). The focus on catalytic wastewater remediation raises awareness of sustainable solutions for global water security (SDG 6). Using a free LLM democratizes high-level research expertise, lowering socioeconomic barriers to STEM entry (SDG 10). Acting as an on-demand virtual teaching assistant, the AI bridges knowledge gaps, offers personalized pacing, and step-by-step breakdowns to accommodate diverse learning needs. To ensure academic integrity, students analyze AI use logs, promoting transparent and ethical prompt engineering practices.