Exemplar research design

Iron leaching from spinach

Context for this exemplar work:
This exemplar *Research design* section is from a student who received a moderated IA grade of 20 out of 24. This indicates that the research design section is likely to be quite good in terms of the structure, relevance and depth of scientific ideas.

Research design exemplar

How does the length of time over which spinach is heated in 1.00 mol dm^{-3} sulfuric acid at 80 °C affect the amount of Fe^{2+} ions leached into the sulfuric acid solution as measured using a redox titration with potassium permanganate?

Introduction:

I chose this topic for research because it relates to the nutritional value of food after heating. Iron is important for oxygen transport in the blood, the immune system, and energy production.

Research question:

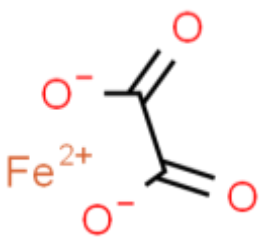
How does the length of time over which spinach is heated in 1.00 mol dm^{-3} sulfuric acid at 80 °C affect the amount of Fe^{2+} ions leached into the sulfuric acid solution as measured using a redox titration with potassium permanganate?

Background information:

Oxalic acid is an organic compound found in many plants, leafy greens, vegetables, fruits, nuts, seeds, and cocoa. For example, vegetables such as spinach are rich in iron (II) ("Oxidation-Reduction Reactions"). Fe^{2+} ions in spinach could be founded bound to non-heme enzymes involved in oxidation-reduction reactions and the transfer of electrons (Ems et al.).

The oxalic acid is usually bonded to minerals, forming oxalate, which can form compounds once consumed, including calcium oxalate and iron oxalate ("Oxalate (Oxalic Acid)"). Even though vegetables like spinach have iron in them, which oxalic acid binds with, people can still consume the amount of iron needed when some of it is broken down in the body. The bonds are broken through boiling vegetables so the iron embedded with oxalic acid can be leached. However, this might cause people to consume less amount of iron as all the iron in the spinach will

Figure 1



be leached into the water left. Figure 1 ("Oxalate (Oxalic Acid)") depicts the compound formed by Fe^{2+} and oxalate ion.

Redox titrations use an oxidation-reduction reaction to calculate the concentration of an unknown solution. The redox process can be oxidation, where a metal element reacts to form a compound and the metal atoms lose electrons to form an ion, or it can be a reduction, where the compounds react for metal elements to be formed, gaining electrons ("Redox Reactions - Metals - National 5 Chemistry Revision - BBC Bitesize").

The equation used in this experiment is:

$$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow Mn^{2+} + 4H_2O + 5Fe^{3+}\text{(Lavelle)}$$

Since potassium permanganate is a pink chemical and Mn^{2+} is colorless, there is no need for an indicator as it involves a color change to pink at the end. Oxidation-reduction (redox) reaction is a chemical reaction where electrons are transferred between two species. Oxidation occurs when a loss of electrons from a substance is involved in a redox reaction. On the other hand, the gain of electrons is called reduction ("13.1: Oxidation-Reduction (Redox) Reactions"). Oxidation and reduction occur together since any loss of electrons by one substance must be accompanied by a gain in electrons. The reactants in redox reactions can be described as oxidizing and reducing agents that transfer electrons between reactants, forming products. An oxidizing agent, or oxidant, is the electron acceptor since it gains electrons and is reduced in a chemical reaction, such as Potassium Permanganate ($KMnO_4$). A reducing agent or reductant is known as the electron donor since it loses electrons and is oxidized in a chemical reaction, such as iron (Fe) ("Oxidizing And Reducing Agents"). A redox titration could be used in this experiment because spinach has non-heme iron (II) that is strongly attached to proteins, which are involved in redox reactions, allowing redox titration to be used to find the amount of iron leached. A solution forms when a solute dissolves in a solvent ("Concentration Of Solutions - BBC Bitesize"), and concentration is the measure of how much of a given substance there is mixed with another substance ("Concentration Terms - SEASTAR CHEMICALS").

Existing literature on the impact of heat on nutrient content suggests that cooking vegetables in low heat is recommended to minimize the loss of nutrients since their content reduces due to leaching in the water (Spritzler). Another experiment looks at the losses of minerals in foods when cooked and their nutritional significance. The results showed that the mineral contents of cooked foods were about 60 to 70% lower than the mineral contents of raw foods. The most significant loss in mineral content was seen in vegetables.

Variables:

Independent variable and values used	Method for obtaining the 5 IV values?
Different time periods where the spinach was heated in sulfuric acid: 5, 10, 15, 20, 25 minutes.	This was measured using a stopwatch.

Dependent variable and units	What data will be collected? How will it be converted to the relevant values/units?
The volume of 0.0500 mol dm^{-3} $KMnO_4$ required to fully react with samples of the Fe^{2+} solution	The volumes will be measured through a redox titration. The volumes of $KMnO_4$ will be converted to moles of Fe^{2+} and then mass of Fe^{2+} per g of spinach.

Controlled Variables	Reasoning
The same volume of sulfuric acid (50 cm^3) was added to the 250 cm^3 beaker in the beginning.	- So that the amount of acid would not have an impact on the leaching of iron while spinach is boiling. - The amount of sulfuric acid needs to be constant to ensure that all spinach is in contact with the acid solution.
The same volume of sulfuric acid (25 cm^3) was added to the conical flask for titration.	- If this variable was not controlled, it could impact the amount of $KMnO_4$ added that fell within the range of the burette volume, affecting the titration.
The same volume of spinach extract solution (10 cm^3) was added to the conical flask for titration.	- The volume was kept constant to make sure that the amount of $KMnO_4$ added always fell within the range of the burette volume.
The boiling temperature was kept at 80 ± 5°C and a thermometer (± 0.5°C) was used to observe the temperature change.	- If the temperature stayed at 100 °C, it could have affected the iron concentration in the spinach as more Fe^{2+} ions would be leached into the sulfuric acid solution due to the bonds of oxalic acid breaking. - The use of a thermometer allowed a more accurate observation of the temperature change and seeing the changes in the solution at different temperatures & times.
The solution was stirred every 30 seconds with the thermometer for all.	- This enabled a more accurate reading of the spinach's temperature
The potassium permanganate was read on the meniscus.	- This was done because it is harder to measure the bottom of the meniscus and identify the volume with darker solutions, such as $KMnO_4$.
A white tile was put under the conical flask during titration.	- This was done to see the color change more clearly and identify the pink color when it has just started changing. This is essential to identify the volume where

	the solution remains in a soft pink color to have a more accurate result for the concentration. If it is not controlled, it could impact the concentration data for each heating time measured.

Uncontrolled variables	Why can it not be controlled effectively?
The size of the spinach pieces	This was too difficult to control due to uneven shapes of spinach leaves. They were cut into approx 1 cm^3 squares and any differences were considered negligible.
Evaporation of water during heating	This could not be controlled but was accounted for by adding the required volume of sulfuric acid before titration of each sample.

Pre-trials:

Before starting the experiment, which was adapted from a redox titration experiment (Royal Society Of Chemistry), a pre-trial was conducted to identify sources of error, including the control and uncontrolled variables that could impact the experiment's findings.

- I added the spinach and sulfuric acid in the beginning and waited for them to heat together. Nevertheless, I realized that the spinach was being heated for longer than the allotted time as it took time to reaching 80°C. So, the sulfuric acid was heated, and the spinach was added when the sulfuric acid reached the desired temperature. Therefore, my research question before the pre-trials was to investigate the heating time, starting from 5 minutes.
- I decided to use 15 g of spinach because it was enough spinach to heat with sulfuric acid at the chosen time periods and collect the filtrate. After collecting the filtrate, the volume was made up to 100 cm^3 to ensure water loss was taken into account during heating.
- Lastly, since my new research question varied by 5 minutes, the potassium permanganate was diluted by 50%. This was done by putting 100 ml of the potassium permanganate and 100 ml of distilled water in a conical flask.

Equipment:

Equipment	
Electronic balance (±0.001 g)	250 cm^3 conical flask (±0.15 ml)
250 cm^3 beaker	50 cm^3 burette (±0.05 ml)
50 cm^3 measuring cylinder (±0.1 ml)	10 cm^3 pipette (±0.05 ml)
25 cm^3 measuring cylinder (±0.1 ml)	Pipette filler

100 cm^3 measuring cylinder (±0.5 ml)	Analog thermometer (±0.5 °C)
Hot plate	100 cm^3 volumetric flask (±0.08 ml)
Buchner funnel	
Chemicals	
2.5 dm^3 of 1.00 mol dm^{-3} sulfuric acid (H_2SO_4)	250 g of spinach
200 cm^3 of 0.0100 mol dm^{-3} potassium permanganate ($KMnO_4$)	100 cm^3 of distilled water

Procedure:

1. Add 50.0 ±0.1 cm^3 of 1.00 mol dm^{-3} sulfuric acid using a measuring cylinder.
2. Pour the sulfuric acid into a beaker and wait for it to boil.
3. Cut the spinach leaves into around 1 cm small pieces and place them in a 250 cm^3 beaker.
4. Weigh 15.000 ±0.001 g of spinach using an electronic balance .
5. Put the spinach leaves into the beaker with sulfuric acid and heat for 5 minutes at 80.0 ± 0.5 °C.
6. Allow the mixture to cool for 10 minutes and filter it by placing a filter paper on the Buchner funnel and using vacuum filtration.
7. Wash the residue in the funnel once with a little distilled water and collect the filtrate. This was done to ensure all solution was being filtered.
8. Pour all the filtrate and the washings into a 100 ±0.08 cm^3 volumetric flask. Makeup to the mark with 1.00 mol dm^{-3} sulfuric acid. This was done to take water loss during heating into account.
9. Put a stopper on the flask and shake it. The flask was shaken to make sure the sulfuric acid and filtrate were mixed.
10. Add 100.0 ±0.5 cm^3 of 0.0100 mol dm^{-3} potassium permanganate solution and 100.0 ±0.5 cm^3 of distilled water into a conical flask to form 200 cm^3 of 0.0050 mol dm^{-3} of $KMnO_4$ solution.
11. Fill a burette with the diluted potassium permanganate solution to the 0.00 cm^3 mark.
12. Using a pipette, transfer 10.00 ± 0.05 cm^3 of the spinach extract solution to a 250 cm^3 conical flask and add 25.0 ± 0.1cm^3 of 1.00 mol dm^{-3} sulfuric acid using a measuring cylinder. This dilution allowed for greater differences in the amount of potassium permanganate added.
13. Titrate the solution into the conical flask with 0.0100 mol dm^{-3} potassium permanganate solution from the burette until the pink color remains for 30 seconds. The pink color had to remain for 30 seconds as the color started to fade away until 30 seconds was completed.
14. Repeat steps 1-13 three times so that I can minimise the impact of random error by calculating an average.

15. Repeat steps 1-14 for all heating times (5, 10, 15, 20, and 25 minutes)

Safety and environmental issues:

Chemical/procedural step	Hazard	Precaution
1.00 mol dm^{-3} sulfuric acid (Fishersci.Co.Uk, 2020)	- Irritant to skin and eyes.	- Wear goggles. - Wash exposed skin or rinse eyes thoroughly.
0.01 mol dm^{-3} potassium permanganate (Fscimage.Fisherci,Com, 2008)	- Irritant to skin and eyes. - Skin contact can cause brown stains in the area.	- Wash exposed skin or rinse eyes thoroughly. - Was clothing before reuse if contaminated
Boiling sulfuric acid	- In this procedure it will be close to boiling and so prevents an additional burn hazard.	- Wear goggles and take care when stirring spinach.

Environmental Considerations: There are no environmental concerns for this experiment as all reagents do not cause environmental harm, so they can be disposed of in a bin or down the sink.