Optimization of Electrochemical Reduction Using S.S Vials Via Mechanochemistry

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Electrochemistry

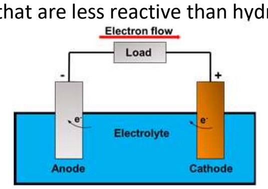
-Two different conductive metals immersed in an electrolyte solution

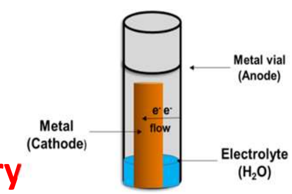
-Electric current is generated due to the oxidation-reduction reaction

-In a galvanic cell, one metal undergoes oxidation (anode) while the other undergoes reduction (cathode)

-The flow of electrons in an electrochemical cell depends on the identity of the reacting substances, the difference in the potential energy of their valence electrons, and their concentrations[1]

-Metals can be ordered into activity series where metals that are more reactive than hydrogen act as reducing agents while metals that are less reactive than hydrogen act as oxidizing agents[2]





Mechanochemistry

-Chemistry is achieved through use of machines

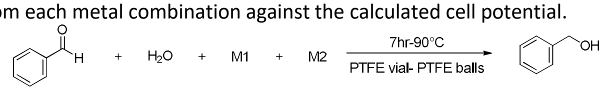
-Instead of solution reactions done in beakers, they are done in vials that are made of stainless steel loaded with stainless steel balls to increase energy of collisions when shaken by the mill

-Advantages: Safety, Environmental Benefits from reduced solvent waste[3]

-The presence of the stainless-steel metal vial and stainless-steel ball with water as electrolyte allow for the merge between mechanochemistry and electrochemistry.

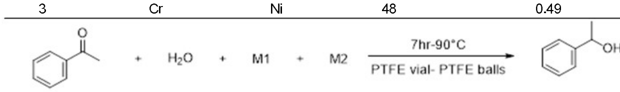
Project Objective

In this research, the metals in stainless steel were being tested for their ability to reduce the substrate successfully. The use of stainless-steel metal components electrodes would give an idea of which metals are responsible for reducing organic substrates. Stainless steel is composed mainly of iron, chromium, and nickel. Instead of using S.S vials, PTFE vials/balls were used to eliminate the interference of S.S metal components and to be able to use metal combinations in equal ratios of metals. The use of PTFE vial and balls allow for observation of the role of each of the metals. These metal combinations were tested for the reduction of benzaldehyde (1a) and acetophenone (1b) to alcohol form. The results show the % conversion from each metal combination against the calculated cell potential.



1a 2a

Entry	M1	M2	% Conversion 1a	Calculated cell potential (V)
1	Fe	Cr	>99	0.3
2	Fe	Ni	>99	0.19
3	Cr	Ni	48	0.49



1b 2b

Entry	M1	M2	% Conversion 1a	Calculated cell potential (V)
1	Fe	Cr	5	0.3
2	Fe	Ni	>99	0.19
3	Cr	Ni	4	0.49

Conclusion

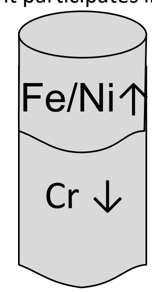
-Merge between electrochemistry principles with a mechanochemical methodology of high-speed ball milling allowed for the ability to overcome the limitation of each one and provide a new tool to carry out chemical reactions

-Data for the benzaldehyde reactions (1a) shows that conversion to final product follows the trend of cell potential

-Data for the acetophenone reactions (1b) shows that this trend is suppressed by the chromium when it participates in the metal combination

-This means the reaction vial that is used to achieve electrochemical reduction could be optimized by having lower ratios of chromium present with higher ratios of iron and nickel

-Would allow for greater yield of product in electrochemical reactions



References

1. Schotten, C., Nicholls, T. P., Bourne, R. A., Kapur, N., Nguyen, B. N., Willans, C. E. Making electrochemistry easily accessible to the synthetic chemist. *Green Chem.*, ***2020***, *22*, ***3358-3375***
2. Hoon, T., Goh, N., & Chia, L. Studying activity series of metals. *Journal of Chemical Education* **1995** *72(1), 51.*
3. Andersen, J. & Mack, J. Mechanochemistry and organic synthesis: from mystical to practical. *Green Chem.*, **2018**, *20*, 1435-1443

Acknowledgments

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