Christ Journal of Science
www.christcollegerajkot.edu.in
Vol 1/Issue1/ June-December/2025

Strategic optimization of methyl orange synthesis: insights into reaction dynamics and purity enhancement

Kuldeepsinh Jadeja[1], Ridhima Gupta[1], Archi Khant[1], Riddhi Joshi[1] Hemangi Rahod[1], Nimisha Joseph[1], Shailesh Thakrar[1], Yvonne Fernandes[1], Abhay Bavishi[1]*

[1]*Christ College, Rajkot*
dr.abhay@christcollegerajkot.edu.in

Abstract:

Methyl orange, an essential azo dye, is widely used in various industrial and laboratory applications due to its distinct pH-sensitive color properties. This study investigates the synthesis of methyl orange through diazotization of sulfanilic acid and subsequent azo coupling, with a focus on optimizing sodium hydroxide (NaOH) concentration and reaction temperature. Experiments varying NaOH concentrations (20–60%) and temperatures (70–90°C) revealed that the highest yield **(95.21%)** was achieved at 60% NaOH and 70°C. UV-visible spectroscopy confirmed the purity of the synthesized dye, with absorbance values closely matching the standard (-0.105 at 600 nm). These findings underscore the importance of precise control over reaction parameters for scalable and efficient azo dye synthesis, offering valuable insights for industrial applications.

Keywords: Azo Dyes, Methyl Orange, Diazotization, Azo Coupling, Sodium Hydroxide Optimization, Reaction Dynamics, UV-Visible Spectroscopy

Introduction

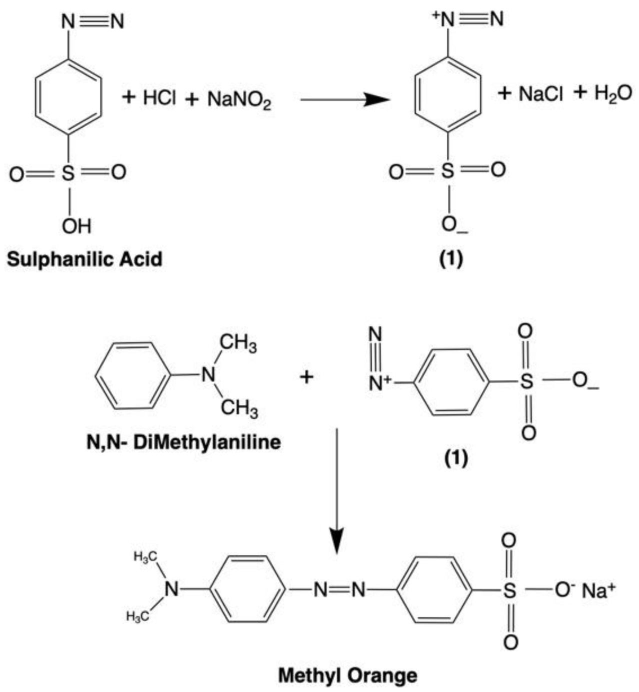
Azo dyes are among the most versatile and widely used synthetic dyes, characterized by their azo (-N=N-) functional group. These dyes are extensively employed in industries such as textiles, cosmetics, food processing, and pharmaceuticals due to their vivid colors, chemical stability, and cost-effectiveness [1,2]. Methyl orange, a prominent member of this class, is particularly valued for its pH-sensitive color changes, making it a critical component in analytical chemistry and laboratory applications [3,4].The synthesis of azo dyes typically involves a two-step process: diazotization of an aromatic amine and subsequent azo coupling with an electron-rich coupling partner. Reaction parameters such as pH, temperature, and reagent concentration critically influence the efficiency, yield, and purity of the resulting dye [5,6].

Despite the established simplicity of this process, optimizing reaction conditions remains a significant challenge, particularly for scaling production while maintaining product quality [7].Previous studies have explored various methodologies to enhance the synthesis of azo dyes, including modifications in reaction pathways, the use of alternative reagents, and the development of eco-friendly processes[8,9]. However, limited research has systematically investigated the interplay between sodium hydroxide (NaOH) concentration and reaction temperature in the synthesis of methyl orange. Such parameters are crucial, as they directly affect the diazotization and coupling reactions, determining both the yield and the purity of the final product.

This study addresses this gap by systematically optimizing NaOH concentration and reaction temperature during methyl orange synthesis. The purity and consistency of the synthesized dye were assessed using UV-visible spectroscopy, ensuring alignment with standard absorbance values. By providing a detailed analysis of reaction dynamics under varying conditions, this research aims to offer a scalable and efficient framework for industrial methyl orange production[10,11].

Experimental Procedure

Chemical Reaction



Materials:

Sulfanilic Acid: 2.1 g per reaction, Sodium Carbonate (Na_2CO_3): 0.46 g, Sodium Nitrite ($NaNO_2$): 0.74 g, Hydrochloric Acid (HCl): 2.2 mL, Sodium Hydroxide (NaOH): 20%, 30%, 40%, 50%, 60% solutions, Sodium Chloride (NaCl): 3 g

Methodology

Diazonium Salt Preparation: Sulfanilic acid was dissolved in water with sodium carbonate, followed by the addition of sodium nitrite. Hydrochloric acid was introduced dropwise under controlled temperatures to form the diazonium salt. **Azo Coupling:** The diazonium salt was reacted with a coupling agent in the presence of NaOH solutions of varying concentrations (20%–60%) at three different temperatures (70°C, 80°C, 90°C).**(Table-1) Isolation and Purification:** The precipitated product was isolated using NaCl, filtered, and recrystallized with distilled water.**Analysis:** The yield was calculated as a percentage of theoretical mass, and UV-visible spectroscopy was performed at 600 nm to assess the purity.**(Fig.1)**

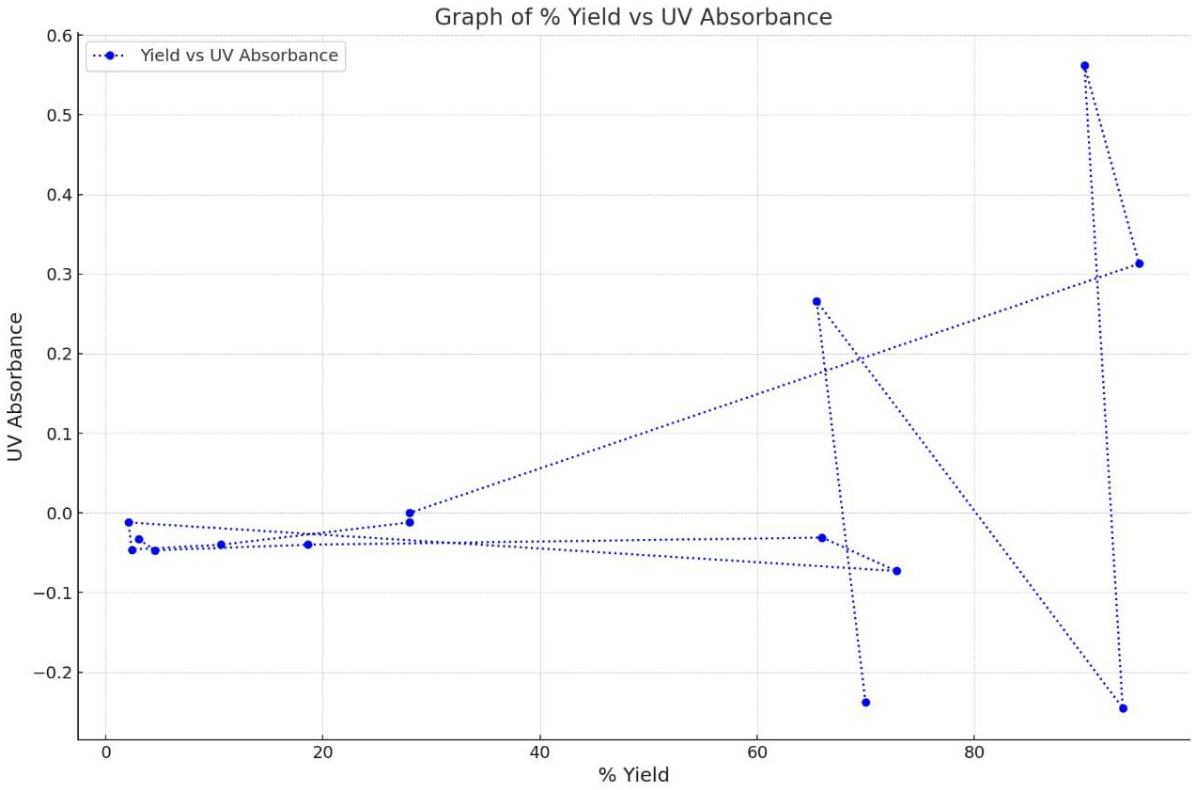
Table 1

Sr No.	Sulphanilic Acid (g)	NaOH	Temperature	Methyl Orange	% Yield	UV Absorbance
1	2.1	20	70	2.79	70	-238
2	2.1	30	70	2.6	65.5	0.266
3	2.1	40	70	3.72	93.7	-0.245
4	2.1	50	70	3.58	90.17	0.562
5	2.1	60	70	3.78	95.21	0.313
6	2.1	20	80	1.113	28	0.300
7	2.1	30	80	1.1	28	-0.012

8	2.1	40	80	0.42	10.6	-0.040
9	2.1	50	80	0.095	2.39	-0.046
10	2.1	60	80	0.082	2.06	-0.012
11	2.1	20	90	2.87	72.84	-0.073
12	2.1	30	90	2.62	66	-0.031
13	2.1	40	90	0.74	18.6	-0.040
14	2.1	50	90	0.18	4.5	-0.047
15	2.1	60	90	0.12	3.02	-0.033

Figure 1

1. **Effect of NaOH Concentration on Yield:**
   Plot a line graph showing NaOH concentration (x-axis) vs. % Yield (y-axis), highlighting the maximum yield at 60% NaOH.
2. **Effect of Temperature on Yield:**
   Create a bar graph with temperature (x-axis) and % Yield (y-axis) for 70°C, 80°C, and 90°C under 60% NaOH conditions.
3. **UV Absorbance Analysis:**
   A comparative bar graph showing the UV absorbance values of synthesized methyl orange against the standard (-0.105).



Result and Discussion

Effect of NaOH Concentration: The data indicate a strong positive correlation between NaOH concentration and methyl orange yield up to 60%. At 70°C, the highest yield (95.21%) was obtained with 60% NaOH. Lower concentrations (20%–40%) resulted in suboptimal coupling, whereas higher concentrations likely caused side reactions, reducing the overall yield.

Effect of Temperature: Temperature significantly influenced the reaction dynamics. At 70°C, yields were highest across all NaOH concentrations tested. Increasing the temperature to 80°C and 90°C led to a marked decline in yield, likely due to thermal decomposition of intermediates and by product formation.

Purity Assessment: UV-visible spectroscopy confirmed the purity of the synthesized methyl orange. The absorbance at 600 nm (-0.105) closely matched the standard value, validating the reaction conditions. Deviations observed at non-optimal conditions were attributed to incomplete reactions or the presence of impurities.

Implications: These findings highlight the critical role of NaOH concentration and temperature in azo dye synthesis. The results align with theoretical expectations, demonstrating that precise control of reaction parameters is essential for optimizing yield and purity. This study provides a scalable model for industrial synthesis of methyl orange and potentially other azo dyes.

Conclusion

This study successfully optimized the synthesis of methyl orange by varying NaOH concentration and reaction temperature. The optimal conditions—60% NaOH and 70°C—yielded 95.21% of high-purity methyl orange. UV-visible spectroscopy validated the quality of the product, underscoring the importance of controlled reaction parameters. The insights gained from this research offer a robust foundation for scaling up the process in industrial settings. Future research could explore the use of eco-friendly reagents and alternative coupling agents to enhance sustainability.

Acknowledgment

The authors wish to express their heartfelt gratitude to Dr. Sushmita Ganguly, Head of the Department of Chemistry, Christ College, Rajkot, for her invaluable guidance and encouragement throughout this study. Special thanks to Mr. Bhavesh Kyada and Ms. Trupti Valera, Department of Chemistry, for their technical support and insights that significantly contributed to the experimental work. We extend our appreciation to Fr. Dr. Jomon Thomanna, Director of Christ Campus, for his leadership and support. Finally, we acknowledge Christ College, Rajkot, for providing the infrastructure and facilities necessary for conducting this research.

References

[1] Synthesis of methyl orange from sulphanilic acid. https://doi.org/10.13140/RG.2.2.10572.49288.

[2] Benkhaya, S. *et al.* (2020). Classifications, properties, recent synthesis and applications of azo dyes. *Heliyon*. https://doi.org/10.1016/j.heliyon.2020.e03271.

[3] Aljamali, N.M. (2015). Review in Azo Compounds and its Biological Activity. *Biochemistry & Analytical Biochemistry*. https://doi.org/10.4172/2161-1009.1000169.

[4] Zhao, R. *et al.* (2011). One step synthesis of azo compounds from

nitroaromatics and anilines. *Tetrahedron Letters*. https://doi.org/10.1016/j.tetlet.2011.05.054.

[5] Siddique, A.B. *et al.* (2024). Thermodynamic and kinetic insights into azo dyes photocatalytic degradation on biogenically synthesized ZnO nanoparticles and their antibacterial potential. *Heliyon*. https://doi.org/10.1016/j.heliyon.2024.e40679.

[6] Alsantali, R.I. *et al.* (2022). Miscellaneous azo dyes: a comprehensive review on recent advancements in biological and industrial applications. *Dyes and Pigments*. https://doi.org/10.1016/j.dyepig.2021.110050.

[7] Shukla, C.A. *et al.* (2021). Towards sustainable continuous production of azo dyes: possibilities and techno-economic analysis. *Green Chemistry*. https://doi.org/10.1039/D1GC01133B.

[8] Akinyemi, A. *et al.* (2024). The role of catalyst in the adsorption of dye: Homogeneous catalyst, heterogeneous catalyst, and advanced catalytic activated carbon, critical review. *Desalination and Water Treatment*. https://doi.org/10.1016/j.dwt.2024.100780.

[9] JP2007533801A.

[10] Velasco, M.I. *et al.* (2011). A green alternative to synthetize azo compounds. *Dyes and Pigments*. https://doi.org/10.1016/j.dyepig.2010.12.009.

[11] Gudelj, I. *et al.* (2011). Azo Dyes, Their Environmental Effects, and Defining a Strategy for Their Biodegradation and Detoxification. *Archives of Industrial Hygiene and Toxicology*. https://doi.org/10.2478/10004-1254-62-2011-2063.