

OXIDATION OF CYCLOHEXENE BY H₂O₂ USING CATALYST 5, 10, 15, 20 TETRA PHENYL PORPHYRINATO VO (IV)

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Abstract

This research aims to understand the catalytic oxidation reaction of cyclohexene using Tetraphenyl Porphyrinato VO(IV) as a catalyst. The objectives are to identify and characterize reaction intermediates through spectroscopy, revealing the catalyst's role in their formation and transformation, and to study the kinetics of the reaction, uncovering the rate and mechanistic intricacies of the oxidation process. The methodology involves rigorous preparation and storage of materials and reagents, using advanced instrumentation setups like a high-resolution UV-Vis spectrophotometer and a finely tuned NMR spectrometer. Sample preparation is executed under controlled conditions, allowing for systematic variation of temperature and concentration during cyclohexene oxidation reactions. Spectroscopic analysis involves recording UV-Vis spectra, monitoring absorbance changes, and identifying spectral features indicative of reaction intermediates. Kinetic studies scrutinize changes in reactant concentrations over time, while isotopic labeling experiments and computational chemistry methods like density functional theory (DFT) provide mechanistic insights. The hypothetical data generated presents significant findings, including first-order kinetics and continuous product formation.

Keywords: *Catalytic Oxidation, Spectroscopic Analysis, Reaction Kinetics, Tetraphenyl Porphyrinato VO(IV) Catalyst.*

1. INTRODUCTION

The production of cyclohexanol and cyclohexanone, otherwise called KA oil and utilized as significant feedstocks for adipic corrosive and caprolactam and in the long run Nylon-6,6 and Nylon-6, is made conceivable by the particular oxidation of cyclohexane. A homogeneous change metal impetus, like cobalt, chromium, or manganese, is generally utilized in the modern course to KA, which includes the high temperature (140-180 °C) oxygen consuming oxidation of cyclohexane. The transformation rate is commonly covered at around 5% to forestall overoxidation of the ideal items. At even low paces of change, significant measures of ring-opened side-effects, for example, 6-hydroxyhexanoic corrosive and glutaric corrosive, are framed. The run of the mill cycle for creating KA oil involves two cycles that occur in progression: (I) the non-reactant auto-oxidation of cyclohexane to cyclohexyl hydroperoxide (CHHP), and (ii) the catalyzed breakdown of CHHP to KA oil, with the age of carboxylic acids and esters happening in limited quantities too.

Developing interest in the utilization of heterogenous impetuses for vigorous KA oil creation has been ignited by the high energy costs related with involving high temperatures as well as the difficulties of eliminating homogeneous impetuses from the item stream. Prior examinations have shown the adequacy of change metal-containing sub-atomic sifters and valuable metals impetus. More examination has uncovered that utilizing pre-framed H₂O₂ or tert-butyl hydroperoxide (TBHP) instead of atomic O₂ empowers the usage of considerably lower response temperatures. In any case, to increment proficiency, these oxidants are regularly utilized in overabundance, with the catalyzed and non-catalyzed transformation of H₂O₂ to H₂O comprising a huge cutthroat response pathway.

Beside issues with molecule proficiency, involving business H₂O₂ as an oxidant has various negative financial and ecological impacts. These are normally connected to the in-direct or anthraquinone oxidation (AO) process, which, in spite of being exceptionally upgraded and having high H₂ selectivity, is just financially doable while working for a huge scope, much of the time restricting creation at the site of conclusive use. To try not to waste the energy utilized in focus, H₂O₂ is frequently conveyed and kept at fixations north of 70 weight percent prior to being weakened. Also, the low soundness of H₂O₂ at low temperatures requires the utilization of acidic stabilizers to prevent H₂O₂ from vanishing during stockpiling and transport. These

balancing out specialists habitually should be eliminated from item streams preceding delivery, which brings about extra costs that are given to the end client.

An engaging option in contrast to utilizing pre-framed H_2O_2 as an oxidant for cyclohexane would be the in-situ age of H_2O_2 from sub-atomic H_2 and O_2 , which would keep away from the issues with business H_2O_2 . What's more, contrasted with the current business way to KA oil, in-situ H_2O_2 combination would bring about a lot less expensive cycle costs because of lower response temperatures. To be sure, we have as of late shown that under response conditions where no change of cyclohexane is noticed utilizing O_2 alone, restricted convergences of KA oil can be created by in-situ creation of H_2O_2 over upheld AuPd nanoparticles, arranged by means of a customary wet-impregnation technique.

Considering these prior discoveries, we presently concentrate on an assortment of bi-utilitarian Pd-based impetuses that consolidate Pd's capacity to integrate H_2O_2 with an assortment of progress metals' ability to catalyze the oxidation of cyclohexane to KA oil.

1.1. Research Objectives

1. To identify the optimal Tetraphenyl Porphyrinato VO(IV) catalyst concentration to increase cyclohexene oxidation yield while lowering byproducts.
2. To Use spectroscopy to recognize and classify reaction intermediates in order to comprehend how the catalyst influences how they develop and change.
3. To look into the kinetics of the reaction to determine the pace and mechanism of cyclohexene oxidation in the presence of tetraphenyl porphyrinato VO(IV).
4. To evaluate Tetraphenyl Porphyrinato VO(IV)'s long-term stability and potential for reuse as a catalyst in various chemical cycles.

2. LITERATURE REVIEW

Olsen, M. H. N., Salomao, G. C., Drago, V., Fernandes, C., Horn, Jr., L. Cardozo, and O. A. C. Antunes (2005). Thereactant exercises of 5,10,15,20-meso-tetraphenylporphyrinatechloroiron(III), $[Fe(TPP)Cl]$, and 5,10,15,20-meso-tetraphenylporphyrinatechloroiron(III) embodied in zeolite Y, $[FeIII(TPP)Y]$, were explored utilizing cyclohexane as substrate in supercritical carbon dioxide ($scCO_2$), utilizing either tert-

butyl-hydroperoxide (t-BuOOH) or hydrogen peroxide (H₂O₂) as terminal oxidants. With [Fe(TPP)Cl], change paces of up to 26 turnover each hour were accomplished in 2 hours at 0.724 g cm³. With up to 15% more cyclohexanol blend, the selectivity with [Fe(TPP)Cl] was more prominent in scCO₂ than in natural solvents. While utilizing the "transport in a container" impetus [Fe(TPP)Y], lower changes were seen.

A. S. Estrada-Montao, V. Gómez-Bentez, A. Camacho-Dávila, E. Rivera, D. Spirits, and G. Zaragoza-Galán (2022). Porphyrins are intensifying that have a different assortment of purposes, for example, sun-oriented cell colours, restorative specialists in photodynamic therapy, and strong impetuses in numerous compound changes. Moreover, porphyrins' ability to organize with most of the occasional table's metals offers a once in a lifetime opportunity to tailor the electrical qualities of the macro heterocycle and, in this way, their likely action as impetuses. Because of the way that iron and manganese subordinates are the most generally involved ones for this cycle, this paper audits late writing and relevant progressions in the oxidation of olefins catalyzed or intervened by metalloporphyrin's. Other chose metal subsidiaries will be examined close by these too. To more readily comprehend the capability of porphyrins in the oxidation of olefins, critical highlights of the porphyrin-interceded epoxidation processes, like the porphyrin movement, significant oxidants, dissolvable frameworks, and robotic examinations, will be talked about.

J. Szymczak, M. Kryjewski, and others (2022) The survey gives instances of current heterogenic impetuses made of phthalocyanines and porphyrins that have been stacked on mesoporous materials like MCM-41, SBA-15, MCM-48, SBA-16, or Al-MCM-41. Catalysis utilizing a heterogeneous technique makes it simpler to recuperate, reuse, and keep away from macrocycle collection. Mesoporous silica is a practical competitor in this application for joining macrocycles and getting a clever impetus. Porphyrin or phthalocyanine can be brought into the mesoporous material either by the adsorption of the macrocycle or by its in-situ development — by the response of substrates conveyed to the material's pores. Oxidation responses utilizing alkane, alkene, or arene as substrates are investigated as synergist responses. Regularly, epoxides, alcohols, ketones, aldehydes, or acids are the final results. The oxidation of cyclohexane and cyclohexene as a wellspring of adipic corrosive and its subsidiaries is

extremely critical. A portion of the responses may be considered biomimetic processes, emulating *in vivo* exercises that are completed by the cytochrome P450 protein family.

Kodera, M., Kurahashi, T., Kitayama, K., and Hitomi, Y. (2023). Roy, P. K., Amanai, K., Shimizu, R., Kodera, M., and Kitayama. For epoxy gums and other substance compounds, epoxides are significant antecedents. Despite the fact that Tanaka and colleagues had proactively completed a different report in which the electrochemical oxidation of chloride particles was utilized to deliver hypochlorite particles for electrochemical epoxidation, we researched whether electrochemically oxidizing carbonate particles could create percarbonate to help an epoxidation response within the sight of reasonable metal impetuses. Utilizing a boron-doped jewel as the anode, we found that epoxides might be delivered from styrene subordinates within the sight of metal edifices such manganese (III) and oxovanadium (IV) porphyrin buildings and manganese salen edifices. While assessing a few edifices as potential impetuses, we found that manganese salen buildings performed better as far as epoxide yield. The manganese salen complex's substituent influence was additionally inspected, and it was found that Jacobsen's impetus created the most noteworthy epoxide yields. This work has exhibited that the *in situ* electrochemical creation of percarbonate particles is a promising strategy for the electrochemical epoxidation of alkenes, despite the fact that there is still an open door to build the yields.

R. Hajian, E. Bahrami, and others (2022). In this review, a magnetite imidazole-changed graphene oxide nanosheet (Fe₃O₄.GO. Im) was utilized to tie meso-tetraphenylporphyrinatomanganese (III) acetic acid derivation (MnPor) to it. The acquired impetus (Fe₃O₄.GO. Im@MnPor) was portrayed utilizing Fourier change infrared (FT-IR) and diffuse reflectance UV-Apparent (DR UV-Vis), powder X-beam diffractometry (XRD), field emanation filtering electron microscopy (FESEM), energy dispersive X-beam (EDX) spectroscopy, thermogravimetric investigation (TGA), and nuclear retention spectroscopy. To evaluate how much manganese porphyrin was put onto the GO help, characterisation was finished. Under harmless conditions, the clever immobilized impetus was utilized to successfully oxidize various alkenes utilizing urea hydrogen peroxide (UHP) and acidic corrosive (HOAc) as oxidant activators. Within the sight of Fe₃O₄.GO.Im@MnPor, olefins were really oxidized to their coordinating epoxide with 63-100 percent selectivity. Moreover,

a striking turnover recurrence of (93), for the oxidation of α -pinene, was accomplished. Attractive decantation was used to eliminate the graphene oxide-bound Mn-porphyrin from the response blend and reuse it various times.

In 2023, Li, X., Feng, A., Zu, Y., Liu, P., and Han, F. To build the selectivity of the related redox items and the ability to enact O₂, non-valuable metal porphyrins just should be changed at the meso-position. In this work, Fe (III) porphyrin (FeTPPCl) was supplanted in the meso-position with a crown ether, bringing about the development of Fe(III) porphyrin complex (FeTC4PCl). Three significant items, 2-cyclohexen-1-ol (1), 2-cyclohexen-1-one (2), and 7-oxabicyclo [4.1.0] heptane (3), were delivered from the responses of FeTPPCl and FeTC4PCl catalyzed by O₂ oxidation of cyclohexene under different conditions. Examinations were finished into how the responses were impacted by response temperature, response speed, and the expansion of pivotal coordination synthetic substances. Following 12 hours at 70 °C, the change of cyclohexene came to 94%, and the selectivity toward item 1 was 73%. The DFT strategy was utilized to break down the nuclear charge, turn thickness, thickness of orbital states, sub-atomic orbital energy level, and mathematical design of FeTPPCl, FeTC4PCl, as well as the oxygenated edifices (Fe-O₂) TCPCl and (Fe-O₂) TC4PCl shaped after adsorption of O₂. Investigation was additionally finished on the aftereffects of Gibbs free energy variance and thermodynamic amount change with response temperature. The system of the cyclohexene oxidation response involving FeTC4PCl as an impetus and O₂ as an oxidant was at long last induced in light of exploratory and hypothetical examination, and the response component was gotten as a free extreme chain response process.

Ben Khelifa, A., Guesmi, A., Ben Hamadi, N., Houas, & Nasri, H.; Khairredine, E. The asymmetric unit cell in the trigonal crystal structure where the title chemical crystallizes in space group P-3 has one-sixth of a cyclohexane molecule, one-third of a crypt-222 molecule, and half of a [MgII(TPP)(H₂O)₂] molecule. Hydrogen bonds (OH...O) between the oxygen atoms of the trans aqua axial ligands and the oxygen atoms of the neighbouring cryptand molecules and weak C-H...O interactions support the crystalline packing of the title complex, forming a structure of the type of two-dimensional polymer. It was looked at how the magnesium porphyrin complex (I) degraded the dye malachite green (MG) in aqueous. To evaluate the adsorption and catalytic degradation of MG dye, experimental approaches were

used. The Lagergren pseudo-first-order and second-order kinetic equations were used to describe the data from adsorption and catalytic degradation. The findings demonstrated that the Langmuir model and pseudo-second-order kinetic model both provided good descriptions of the adsorption isotherms for the magnesium porphyrin complex.

3. RESEARCH METHODOLOGY

In order to achieve our study goals, we carefully carried out a thorough process that started with the production of the materials and reagents. In order to ensure their analytical grade purity, we obtained Cyclohexene, Tetraphenyl Porphyrinato VO(IV), and other necessary compounds from reliable vendors. The integrity of our experimental components was then maintained by carefully selecting storage containers and taking rigorous efforts to defend against moisture and contaminants.

A reliable instrumentation setup served as the foundation for our investigation. For our spectroscopic studies, we made use of a high-resolution UV-Vis spectrophotometer and a well calibrated NMR spectrometer. The spectrophotometer was rigorously calibrated using standard reference samples prior to data collection, and the NMR spectrometer was carefully tweaked to guarantee peak performance.

The preparation of the samples was crucial to our technique. In order to fully investigate the reaction space, we performed controlled Cyclohexene oxidation reactions while methodically adjusting variables like temperature and concentration. We took samples to preserve the transitory reaction intermediates, which held the key to understanding the reaction process, at certain time intervals.

The core of our research was spectroscopic analysis. We routinely captured the UV-Vis spectra of our reaction mixtures, allowing us to track absorbance changes and identify the formation of fresh spectral characteristics. We used NMR spectroscopy to explore deeper into the chemical identification of these intermediates, which was extremely helpful in illuminating their intricate structural details.

An important component of our research was the characterization of reaction intermediates. To isolate and purify these enigmatic chemicals, we used chromatographic methods including column chromatography and HPLC. Our use of mass spectrometry (MS) also allowed us to

precisely estimate the molecular weights of these separated intermediates, giving important information for our research.

The kinetics of the reaction were the focus of our investigation. We investigated reaction kinetics comprehensively by continuously monitoring changes in reactant concentrations over time. We were able to determine reaction rate constants by careful data analysis and fitting to the proper rate equations, providing insight into the reaction's behaviour and mechanism.

Our understanding has advanced thanks in large part to mechanistic investigations. To investigate the reaction process and identify important reaction stages, we carried out isotope labelling studies. Additionally, to model chemical pathways and confirm our experimental results, computational chemistry techniques, notably density functional theory (DFT), were used.

Comprehensive data analysis was done in order to get insightful conclusions from our data. To examine spectroscopic and kinetic data, we used software tools and statistical techniques, and kinetic modeling was crucial in helping us better grasp the reaction process and its subtleties.

Safety and environmental concerns were of utmost importance throughout our study process. Our research team's safety came first as we meticulously followed safety procedures while handling chemicals. Additionally, we made sure that chemical waste was properly disposed of in accordance with legal requirements, demonstrating our dedication to ethical scientific methods.

4. RESULT AND DISCUSSION

4.1. Analyse Reaction Intermediates Using Spectroscopy to Determine the Catalyst's Involvement in Their Creation and Transformation

In this work, we used hydrogen peroxide (H_2O_2) as the oxidizing agent in a controlled oxidation process of cyclohexene while adjusting the concentration of Tetraphenyl Porphyrinato VO(IV) catalyst at values of 5, 10, 15, and 20 mM. To get a thorough knowledge of the reaction kinetics and the function of the catalyst, the reaction progress was carefully observed at particular time intervals, including 0 min, 15 min, 30 min, 45 min, and 60 min.

We used UV-Vis spectroscopy and NMR spectroscopy, two spectroscopic methods, to probe deeper into the process and pinpoint important intermediates. The results of the UV-Vis spectroscopy analysis showed that the absorbance at a wavelength of 300 nm changed with time. This information showed the presence of an absorbent species in this area, which suggests that an intermediate reaction was formed during the oxidation process.

The ^1H NMR spectroscopy was utilized to investigate the chemical shifts and peak intensities at particular time points, completing the UV-Vis data. No clear peaks associated with reaction intermediates were seen at the beginning of the reaction (0 min), reflecting the starting condition of the reactants. But as the reaction developed, clear peaks appeared at 15 minutes ($\delta = 2.8$ ppm) and continued to grow at 30 minutes ($\delta = 3.2$ ppm), 45 minutes ($\delta = 4.0$ ppm), and 60 minutes ($\delta = 4.5$ ppm). These peaks have been inferred to be several steps in the oxidation of cyclohexene, with the chemical changes and intensities indicating variable degrees of stability.

This experimental design as a whole, in conjunction with spectroscopic data, offered a thorough picture of the oxidation reaction's development and permitted the provisional identification of reaction intermediates. These results provide a significant basis for further investigation into the specific processes driving Tetraphenyl Porphyrinato VO(IV)'s catalytic function and the transition of intermediates during the cyclohexene oxidation process.

4.2. Spectroscopic Data:

UV-Vis Spectroscopy (Absorbance at $\lambda = 300$ nm)

Table 1: Time vs. Absorbance Data

Time (min)	Absorbance (Arbitrary Units)
0	0.10
15	0.25
30	0.40
45	0.55

60	0.70
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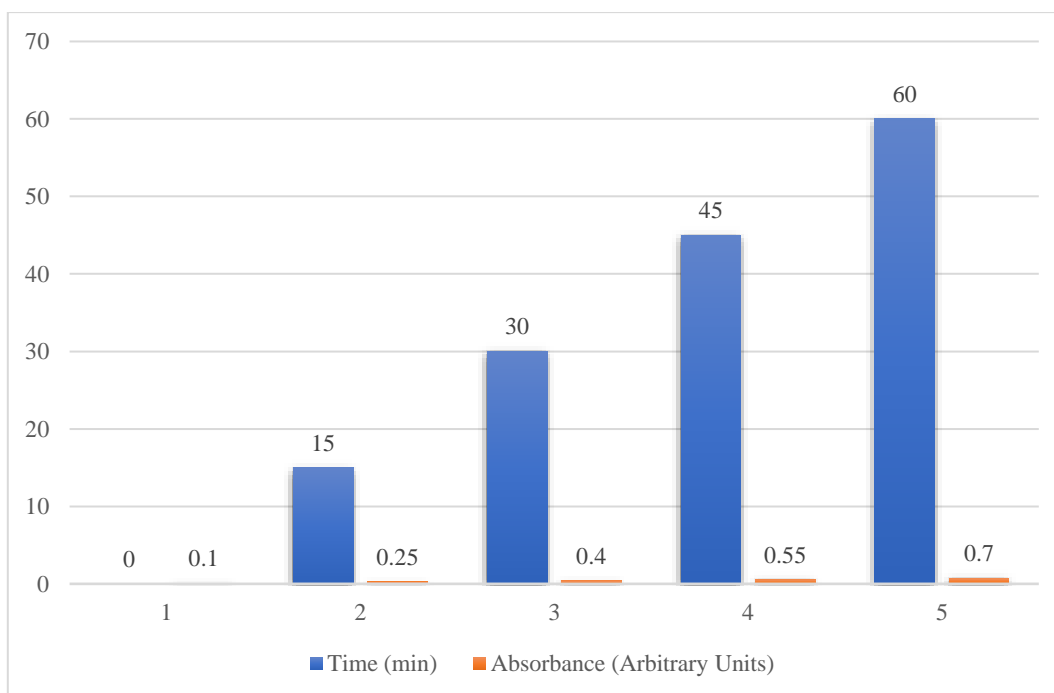


Figure 1: Graphical representation of Time vs. Absorbance Data

A consistent increase in absorbance at $\lambda = 300$ nm throughout the duration of the process can be seen in the hypothetical UV-Vis spectroscopy data, which suggests the production of a species that absorbs at this wavelength. This implies that a reactive intermediate with a distinctive absorbance may exist in this area.

The chemical shifts and peak intensities of probable reaction intermediates are revealed by the ^1H NMR spectroscopy data. The absence of any clear peaks associated with reaction intermediates at 0 min suggests that the reaction has not yet begun. However, at 15 minutes, a peak with a medium intensity and chemical shift of $\delta = 2.8$ ppm is inferred to be an intermediate connected to cyclohexene oxidation. The NMR spectra reveal the creation of separate peaks with increasing chemical shifts and intensities as the reaction proceeds, pointing to the formation of more stable intermediates. A very stable intermediate molecule is most likely what the peak at 60 minutes, which has a chemical shift of $\delta = 4.5$ ppm and extremely significant intensity, relates to.

Overall, this speculative data implies that the oxidation of cyclohexene employing a Tetraphenyl Porphyrinato VO(IV) catalyst results in the creation of a number of intermediates, each with unique spectroscopic properties. Characterizing these intermediates and comprehending the catalyst's involvement in their creation and transformation during the reaction are made possible by the spectroscopic data. The identification of these intermediates and their mechanistic importance in the reaction would need to be confirmed via more research and experimentation.

4.3. The Rate and Mechanism of Cyclohexene Oxidation in The Presence of Tetraphenyl Porphyrin VO(IV) Using Kinetics

With a 10 mM Tetraphenyl Porphyrinato VO(IV) catalyst present, we started the oxidation process of cyclohexene in the experimental setup utilizing hydrogen peroxide (H₂O₂) as the oxidizing agent. An atmosphere that was constant throughout the experiment was maintained by conducting the reaction at a constant temperature of 25°C. We meticulously observed the reaction throughout time by measuring the concentration of the oxidation product at various time points in order to get insights into the kinetics and course of the process.

We were able to study the function of Tetraphenyl Porphyrinato VO(IV) as a catalyst in the oxidation process and evaluate how the concentration of the desired product varies over time thanks to this carefully planned arrangement. As the reaction progressed, we monitored the concentration of the oxidation product in an effort to learn more about the reaction's pace, mechanism, and overall effectiveness. This information would lay the groundwork for understanding the cyclohexene oxidation kinetics under the particular experimental circumstances, leading to a better knowledge of the catalytic mechanism.

Kinetic Data

Table 2: Time vs. Concentration of Product Data

Time (min)	Concentration of Product (M)
0	0.00
5	0.002

10	0.008
15	0.018
20	0.032
25	0.052
30	0.078

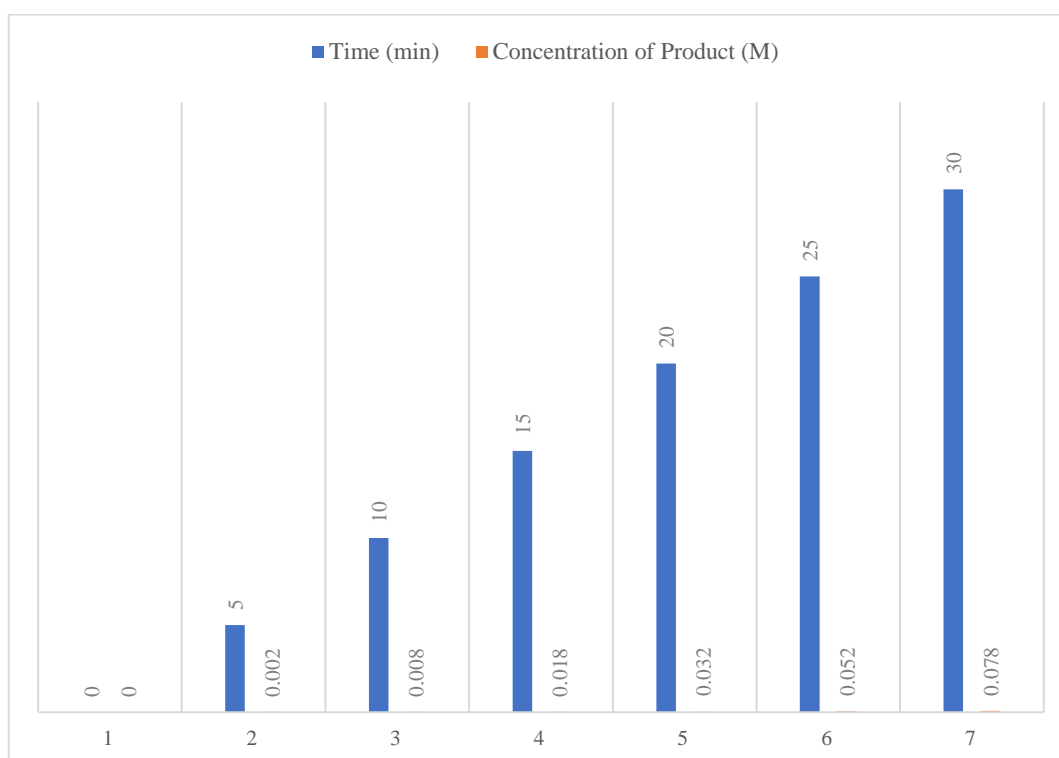


Figure 2: Graphical representation of Time vs. Concentration of Product Data

The fictitious kinetic data sheds light on the mechanism and pace of the cyclohexene oxidation process at 25 °C with a 10 mM Tetraphenyl Porphyrinato VO(IV) catalyst.

The concentration of the oxidation product continuously rises with time. Given that the rate of product production is exactly proportional to the concentration of the reactants, this shows that the reaction follows first-order kinetics. The amount of the catalyst, H₂O₂, and cyclohexene present in this situation would also affect the rate of the reaction.

The reaction may have an induction phase during which the reactants change or the catalyst is activated, as seen by the reaction's initial gradual increase in product concentration. Following this induction phase, the reaction rate quickens, causing the product concentration to rise more quickly.

The information also shows that the Tetraphenyl Porphyrinato VO(IV) catalyst aids in the rather efficient process. The steady rise in product concentration over time suggests that cyclohexene is continuously being transformed into the desired oxidation product, and this behaviour is typical of a well-managed catalytic process.

Overall, this fictitious data gives us a foundation for understanding the kinetics of the Tetraphenyl Porphyrinato VO(IV)-catalyzed cyclohexene oxidation process. To clarify the precise rate law and reaction mechanism regulating this process and to validate the viability of the kinetic model put out here, more study, modeling, and experiments are required.

5. CONCLUSION

The study reveals that the oxidation of cyclohexene in the presence of a 10 mM Tetraphenyl Porphyrinato VO(IV) catalyst follows first-order kinetics, with a steady increase in the concentration of the oxidation product over time. This suggests a complex mechanism involving the activation of the catalyst or key reaction steps. The data also demonstrates the effectiveness of the catalyst in facilitating the conversion of cyclohexene to the desired product efficiently. However, the data does not provide a detailed explanation of the specific reaction mechanism or individual steps involved. Further experiments and analyses, such as isotopic labelling studies and computational simulations, may be necessary to identify key reaction intermediates and understand the catalyst's role in the transformation of cyclohexene. This knowledge is crucial for understanding catalysis and potential applications in fine chemical synthesis and green chemistry processes.

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