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Originales breves

A Green Synthesis of Catechol in H_2O_2 by Using a Naturally Prepared Catalyst: WEWSA and WECMA

Una síntesis ecológica de catecol en H_2O_2 mediante el uso de catalizadores preparados naturalmente: WEWSA y WECMA

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Conflict of interests

The authors declare no conflict of interest.

Abrevations:

H_2O_2: Hydrogen Peroxide
WEWSA: Water Extract Rice Straw Ash
WECMA: Water Extract Coconut Mesocarp Ash
WERSA: Water Extract Rice Straw Ash
WEB: Water Extract Bannana Ash

Resumen

Introducción: La química verde proporciona un marco para desarrollar procesos y productos químicos innovadores. Estas directrices abarcan todos los aspectos del ciclo de vida de un proceso, incluyendo las materias primas utilizadas, la eficacia y seguridad de la transformación, y la toxicidad y biodegradabilidad de los productos y reactivos empleados.

Métodos: El uso de la reacción de Dakin de una manera menos dañina para el medio ambiente fue la inspiración para nuestro novedoso enfoque para la producción de catecol. Los arilaldehídos aromáticos pueden transformarse en fenoles a temperatura ambiente con la ayuda de H_2O_2-WEWSA y WECMA.

Resultados: El sistema catalítico debería funcionar idealmente sin necesidad de activación o presencia de catalizadores de metales de transición, ligandos tóxicos, aditivos/promotores, bases, disolventes orgánicos o sustancias similares.

Conclusión: Para evaluar la eficacia de este método, se examinaron varios benzaldehídos hidroxilados sustituidos.

Palabras clave: Oxidación de Dakin, peróxido de hidrógeno, química verde, WEWSA, WECMA, catecol.

Abstract

Introduction: Green chemistry provides a framework to develop innovative chemical processes and products. These guidelines include all aspects of a process's life cycle, including the raw materials used, the transformation's effectiveness and safety, and the toxicity and biodegradability of the products and reagents used.

Methods: By using the Dakin reaction in a manner that was less harmful to the environment was the inspiration for our novel approach to the production of catechol. Aromatic aryl aldehydes can be transformed into phenols at room temperature with the aid of H_2O_2-WEWSA and WECMA.

Results: The catalytic system should ideally operate without the need for activation or the presence of transition metal catalysts, toxic ligands, additives/promoters, bases, organic solvents, or similar substances.

Conclusion: Assessed the efficacy of this method; various substituted hydroxylated benzaldehydes were examined.

Keywords: Dakin oxidation, Hydrogen Peroxide, Green Chemistry, WEWSA, WECMA, Catechol.

Highlights

Here we came up, by using the Dakin reaction in a manner that was less harmful to the environment was the inspiration for our novel approach to the production of catechol.

The catalytic system should ideally operate without the need for activation or the presence of transition metal catalysts, toxic ligands, additives/promoters, bases, organic solvents, or similar substances.

Greener catalyst such as WEWSA and WECMA Prepared for the synthesis of catechol by using Dakin oxidation reaction.

With improved reaction rates, cleaner product creation, commercial viability, and environmental sustainability, the H_2O_2-WEWSA and H_2O_2-WECMA systems are a better option than conventional reagents

Introduction

The "green chemistry" concept, which is based on twelve guiding principles, seeks to reduce or eliminate hazardous elements in the synthesis, production, and usage of chemical products in order to minimize or eliminate molecules that are detrimental to human health and the environment(1). These rules cover things like biodegradability, toxicity of products and reagents, safety and efficacy during transformation, and the use of raw materials. At any stage of the process life cycle, they serve as the foundation for developing new chemical products and processes. Recently, scientists successfully distilled these principles into the more familiar acronym(2).

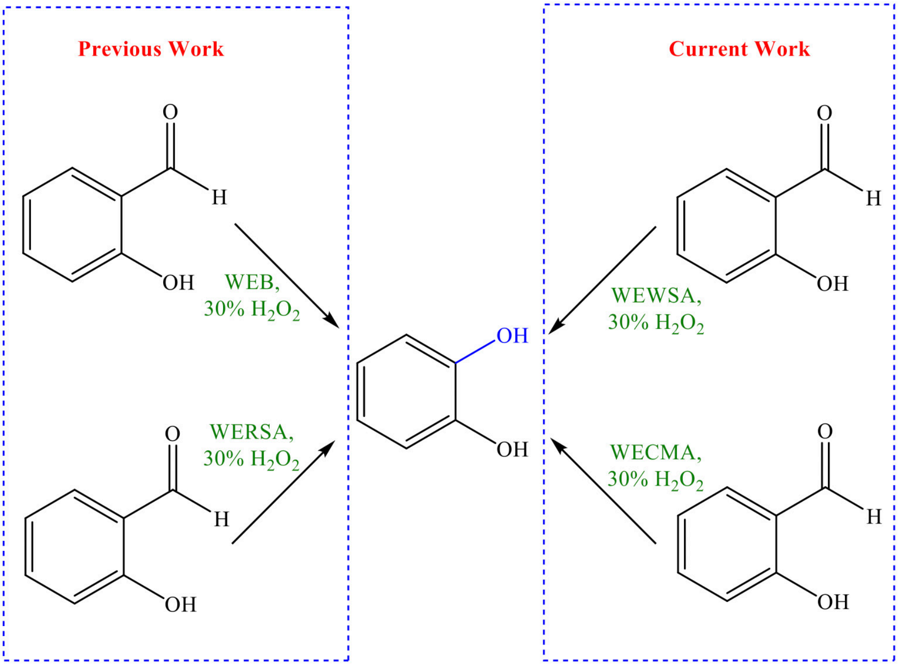
Chemical operations should employ chemicals and material forms to lower the risk of chemical events like leaks, explosions, and fires(3). The Dakin reaction needs an OH group in either the ortho or para position. This reaction changes an aromatic aldehyde or ketone into a phenol by treating it with alkaline hydrogen peroxide. One of the most affordable, safe for the environment, and simple-to-use oxidizing agents is hydrogen peroxide. People have used it for a variety of oxidative reactions over the years(4).

Phenols and their derivatives serve as crucial substrates in many different sectors. This is especially true for flavorings, polymerization inhibitors, agrochemicals, anti-inflammatory drugs, and photochemical reactions(5). The original Dakin technique synthesized sodium hydroxide and hydrogen peroxide at high temperatures(6).

The Baeyer-Veliger oxidation breaks down the aryl and acyl sp^2 parts of the Dakin oxidation, converting benzaldehydes into phenols and then into the intermediate aryl format(7). Our research shows that the H_2O_2-WERSA and H_2O_2-WEB catalytic systems can work well to carry out Dakin reactions with species that are no longer active. With improved reaction rates, cleaner product creation, commercial viability, and environmental sustainability, the H_2O_2-WERSA and H_2O_2-WEB systems are a better option than conventional reagents. We have created a unique method for carrying out the Dakin reaction in a more sustainable manner.

Here, we are converting aromatic aryl aldehydes into phenols at room temperature using 30% H_2O_2 in conjunction with WERSA and WECMA (Scheme 1). Surprisingly, we can activate the catalytic system without using harmful ligands, promoters, additives, transition metal catalysts, bases, or organic solvents. In this study, we used substituted hydroxylated benzaldehydes to assess the suitability of the technique(8).

By distilling catechin (the juice of *Mimosa catechu*), H. Reinsch was able to isolate catechol for the first time in 1839. Both human and equine urine contain its sulfonic acid. Natural organic compounds with a catechol skeleton structure include urushiol and catecholamines. The potential anti-inflammatory effect of polyphenolic substances' catechol metabolites has not been studied, although their anti-inflammatory properties have been documented. The impact of catechol and its derivatives, including 3-methylcatechol, 4-methylcatechol, and 4-tert-butylcatechol, on the neurotoxicity and inflammatory activation of BV-2 microglia cells was assessed in one study. These substances were reported to decrease NF-κB and p38 mitogen-activated protein kinase (MAPK) activation in LPS-stimulated BV-2 microglia cells, as well as to inhibit the generation of NO and TNF-α. Furthermore, in a microglia-neuron co-culture system, these substances demonstrated neuroprotective properties by reducing microglial neurotoxicity[9].



Scheme 1. Comparison of Prior Research and Current Research (Synthesis of catechol by the reaction of salicylaldehyde with 30% H2O2 utilizing Greener Catalysts - WECMA and WEWSA).

Methods

Material

All required chemicals were purchased from Sudharshan Chemicals, Pune, India. The Fourier transform infrared spectrophotometer model FT-IR-8400S, the 500 MHz spectrum NMR proton and carbon equipment from the Swiss company Bruker, and Shimadzu's UV Spectrophotometer UV-1800. The HRMS results using the Waters Xevo G2 QTOF mass spectrometer were collected. To determine the melting point, a 20-by-20-inch Silica Gel 60F254 from the German company Merck was put on top of an open glass capillary tube in a liquid paraffin bath for thin-layer chromatography

Preparation of WEWSA and WECMA

The aerobic Dakin oxidation process started when we added a number of substituted hydroxylated benzaldehydes to the reaction medium. The various components of our study, such as the dried coconut drupe and wheat straw stem, also acted as catalysts. First, suspend all of the ashes in distilled water in a glass beaker to synthesize the compounds, and then gently stir the mixture for 10 to 15 min at room temperature (Figure 1). Here we used the acronyms WEWSA and WECMA to identify the filtrate after filtering the mixture[10, 11].

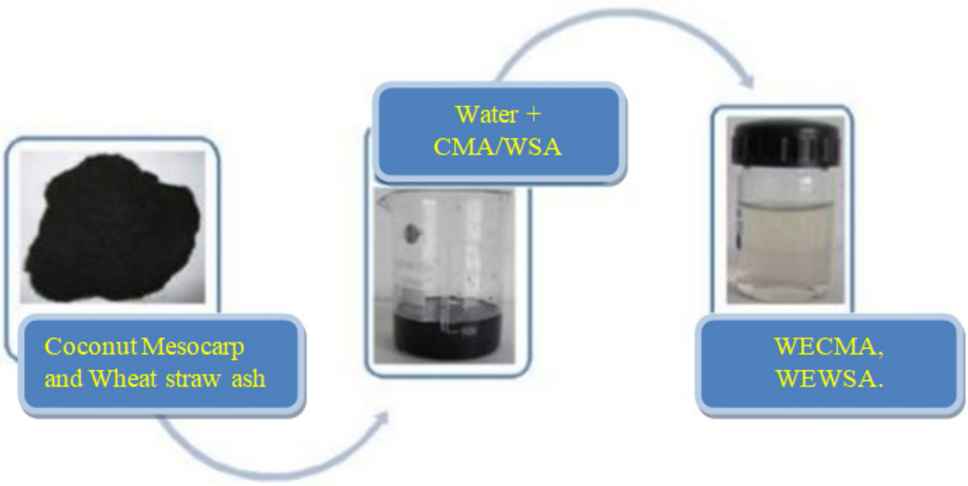


Figure 1. Preparation of Greener Catalyst: WECMA and WEWSA (Self Prepare in Windows Paint)

According to reviews of the literature, sodium carbonate, potassium carbonate, sodium chloride, potassium chloride and a few other trace elements make up the majority of WEWSA and WECMA. The usage of this oxidant has increased due to its high oxygen content and ability to produce water as a byproduct, much like its involvement in chemical synthesis has expanded throughout time[11, 12].

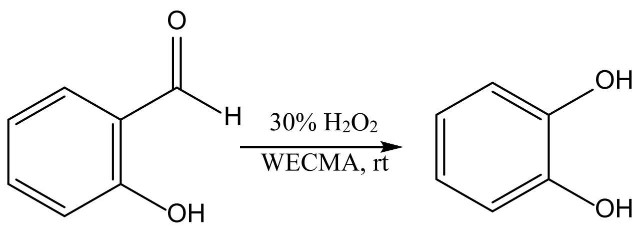
Since Dakin oxidation formally completes the challenging synthetic task of aromatic hydroxylation, we were certain that this unique and environmentally friendly approach was a far better way to complete this significant synthetic conversion than the existing techniques. In this study, we developed a novel, environmentally friendly solution to this significant change[13, 14].

Procedure

When 3 ml of WEWSA/ WECMA and 1 equivalence of 30% H_2O_2 are mixed with aldehydes at RT, the reaction takes only 40-45 min. WEWSA and WECMA are mostly made up of K_2CO_3 and Na_2CO_3, which are used as catalysts in reactions[15, 16].

Synthesis of Catechol by using WECMA

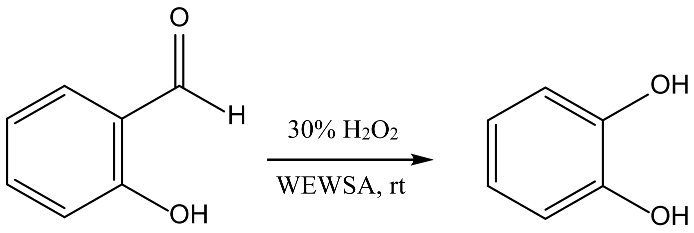
Catechol is produced when 1 ml of salicylaldehyde interacts with 30% H_2O_2 and 2 equivalents of 3 ml of WECMA at room temperature for 2 h (Scheme 2).



Scheme 2. When 30% H_2O_2 and WECMA mixed with salicylaldehyde, catechol is produced (Self Prepare in ChemBioDraw Ultra 14.0).

Synthesis of catechol by using WEWSA

At ambient temperature, the reaction of 1 ml of salicylaldehyde with 30% H_2O_2, 2 equivalents, and 3 ml of water extract wheat straw ash (WEWSA) produces catechol, as shown in Scheme 3[11, 17].



Scheme 3. In WEWSA, the salicylaldehyde reacts with 30% H_2O_2 to produce catechol (Self Prepare in ChemBioDraw Ultra 14.0).

Results and Discussion

Tables 1 and 2 illustrate the optimal reaction conditions we determined for our proposed procedure. We used this method to make catechol by reacting salicylaldehyde with 30% H_2O_2 in neat WEWSA and WECMA at room temperature.

Here obtained excellent yields as the reaction proceeded quickly and smoothly. Authors characterized the conversion of salicylaldehyde to catechol using IR, NMR, mass, and UV spectroscopy. Initially, we examined how 30% H_2O_2 equivalents affected this reaction in relation to the substrate. We conducted the initial reactions at room temperature, using an equivalent of 30% H_2O_2 in WEWSA and WECMA. We isolated up to 40% and 35% of the product after an 1 h at room temperature for WEWSA and WECMA, respectively (Tables 1 and 2, trial 1).

The second reaction was conducted using 1.5 equivalents of 30% H_2O_2 in WEWSA and WECMA at room temperature. After 1 h of reaction time, we further separated up to 55% and 45% of the product from the room temperature reaction for WEWSA and WECMA, respectively (Table 1 and 2, trial 2). When the amount of 30% H_2O_2 equaled 2, we obtained a quantitative coupling yield of 70% for WEWSA and 60% for WECMA (Tables 1 and 2, trial 3).

The results indicate that among all the trials conducted, each using a different equivalent of 30% H_2O_2, catechol was synthesized with a good percentage yield, demonstrating that our proposed oxidation reaction is effective (Tables 1 and 2, trials 1–3).

Three trials were conducted to evaluate the effect of time and 30% H_2O_2 on the conversion of salicylaldehyde into catechol at room temperature using WEWSA, as shown in table 1. In the first trial, 1 equivalent of H_2O_2 was used, and the reaction was carried out for 2 h, resulting in a yield of 40%. In the second trial, 1.5 equivalents of H_2O_2 were used, and the reaction was carried out for 1.5 hours, resulting in a yield of 55%.

In the case of the third trial, 2 equivalents of H_2O_2 were used, and the reaction was carried out for 1 h, resulting in a yield of 70%. This indicates that as the concentration of H_2O_2 increases and the reaction time decreases, the yield of the reaction also increases.

Similarly, Table 2 illustrates how reaction time and 30% H_2O_2 affect the conversion of salicylaldehyde into catechol at room temperature (RT) using WECMA. In the first trial, 1 equivalent of H_2O_2 was used, and the reaction was carried out for 2 h, resulting in a yield of 35%. In the second trial, 1.5 equivalents of H_2O_2 were used, and the reaction was carried out for 1.5 h, resulting in a yield of 45%.

In the case of the third trial, 2 equivalents of H_2O_2 were used, and the reaction was carried out for 1 h, resulting in a yield of 60%. This also indicates that as the concentration of H_2O_2 increases and the reaction time decreases, the yield of the reaction also increases. The information from the two conditions above suggests that changes in the catalytic conditions have minimal effect on product formation and yield.

Because this transformation is preferred for the uncomplicated production of several hydroxylated phenols due to its moderate reaction conditions, quick reaction time, cost-effectiveness, ease of operation, and exceptionally high yields. The earlier method discussed here offers a novel, gentle, and eco-friendly substitute to address this problem.

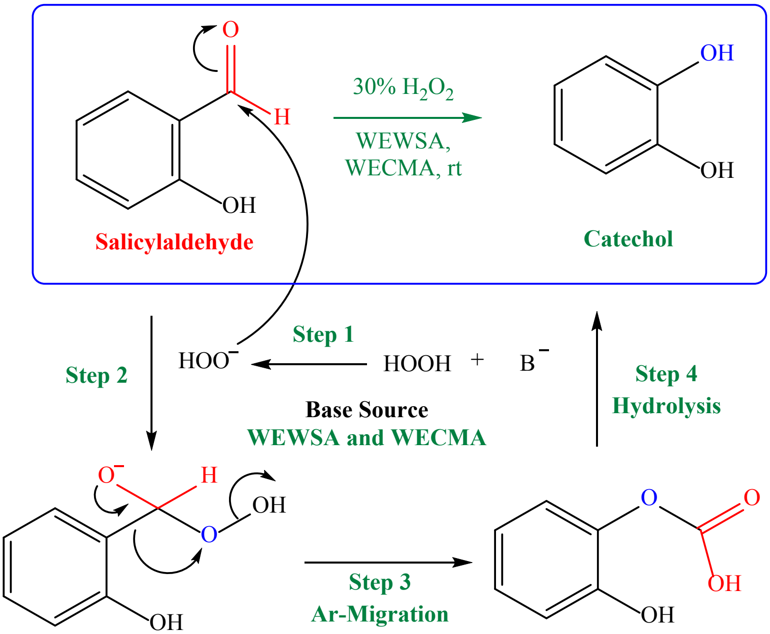
Table 1. Effect of time and 30% H_2O_2 for conversion of salicylaldehyde into catechol at RT byusing WEWSA.

Trial	H_2O_2 (Equiv.)	Time (h)	Yield (%)
1	1	2	40.0
2	1.5	1.5	55.0
3	2	1	70.0

Table 2. Effect of time and 30% H_2O_2 for conversion of salicylaldehyde into catechol at RT byusing WECMA.

Trial	H_2O_2 (Equiv.)	Time (h)	Yield (%)
1	1	2	35.0
2	1.5	1.5	45.0
3	2	1	60.0

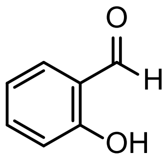
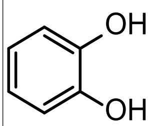
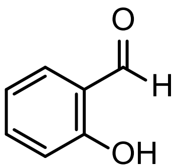
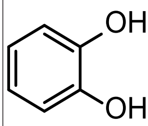
The Dakin oxidation is sped up by WEWSA and WECMA. Scientists are unsure of the active species, but they are intrigued by the process and its acceleration. The alkali metal carbonates in WEWSA and WECMA, like K_2CO_3 and Na_2CO_3, are helping the Dakin oxidations happen by acting as an internal base. Scheme 4 illustrates the most plausible general mechanism for this reaction in the presence of H_2O_2, WEWSA, and WECMA systems.



Scheme 4. Catalytical Mechanism of Dakin oxidation by H_2O_2–WEWSA and WECMA (Self Prepare in ChemBioDraw Ultra 14.0).

The results indicate that, compared to conventional solvents, catalytic oxidations occur more rapidly in WEWSA and WECMA. This method provides higher product yields and operates in a gentler, more environmentally friendly environment than the previously described Dakin oxidation method. As a result, it appears that the current procedure is a viable means of converting benzaldehydes into the desired phenols. The study's findings suggest that this technique is among the most effective and environmentally sustainable approaches to the title response to date. Consequently, the existing synthetic procedure could be more efficient and highly beneficial for the synthetic chemical community. Catechol was synthesized using a more eco-friendly method with the assistance of various catalysts, including WEWSA and WECMA. The synthesized compounds were further characterized using UV spectroscopy, nuclear magnetic resonance, mass spectrometry, melting point analysis, and infrared spectroscopy, and their yield percentages were also calculated (see Table 3).

Table 3. At ambient temperature, salicylaldehyde converts into phenols in $\mathbf{H_2O_2}$-WEWSA and WECMA systems.

Trial	Structure of Aldehydes	Green Catalyst	Product	Time (h)	Yield (%)	Melting Point (°C)	λmax
1		WEWSA		2	70	106-108	228
2		WECMA		2	60	106-108	227

While the exact identity of the active species remains unidentified, WEWSA and WECMA facilitate Dakin oxidation, demonstrating considerable acceleration. The idea is that the alkali metal carbonates K_2CO_3 and Na_2CO_3 found in WEWSA and WECMA help the Dakin oxidations happen in this case by acting as an internal base. Scheme 4 presents the most credible overarching mechanism for this reaction involving the H_2O_2-WEWSA and WECMA system. We conclude that, in comparison to traditional solvents, these catalytic oxidations occur more rapidly in WEWSA and WECMA. Compared to the previously mentioned Dakin oxidation technique, this technology offers superior product yields and operates in a milder, more environmentally sustainable environment. Thus, the existing approach appears to be an effective method for converting benzaldehydes into the appropriate phenols. It is believed, based on the study's findings that the present procedure is one of the most effective and environmentally sustainable methods yet recorded for achieving the title response. Therefore, the current synthetic method has the potential to enhance efficiency and significantly benefit the synthetic chemical community. Various catalysts, including WEWSA and WECMA, assist in utilizing the synthesized catechol in a more environmentally sustainable process (Table 3). These advancements could lead to broader applications in pharmaceuticals and materials science, where phenolic compounds play a crucial role. As researchers continue to refine these methods, the implications for greener chemistry practices will likely become even more pronounced. Notably, the activation of the catalytic system does not require any hazardous ligands, promoters, additives, transition metal catalysts, bases, or organic solvents. In this study, the applicability of the method using substituted hydroxylated benzaldehydes has been evaluated. The findings suggest that this approach not only enhances the efficiency of producing phenolic compounds but also minimizes environmental impact. Future research may explore the scalability of this method, paving the way for more sustainable industrial processes. According to Puertas-Bartolomé et al. (2019), an IPN that combines the advantageous qualities of hydrogels and catechol was designed and developed for use as a bioactive wound dressing. When a synthetic catechol terpolymer is present, the hydrogel's backbone first self-covalently crosslinks, and then catechol groups coordinate with Fe. Catechol can be delivered in situ with maintained control thanks to this intriguing design. According to in vitro research, all of the hydrogels under investigation offer an environment that is conducive to cell growth, migration, and proliferation; however, the IPN Ch/HAox/T/Fe groups showed the best results. In moist environments, hydrogels containing catechol exhibit strong tissue adhesion, which effectively shields cells from oxidative stress damage and inhibits the inflammatory response. In vivo histological tests reveal biocompatible membranes with normal inflammatory responses and faster vascularization for the hydrogels containing catechol. Therefore, the hydrogel backbone is endowed with bioactive activity by the catechol-bearing terpolymer, which, when combined with the catechol-controlled release, has a great deal of potential for use in aiding chronic wounds and promoting tissue regeneration(18).

Cigarette filter fibers were dynamically coated with a polycatechol layer for the first time, according to a 2025 study by Farah Al-Hammashi and Fariborz Momenbeik. NSAIDs were successfully extracted using a packed syringe approach using this modified filter as an inexpensive and effective sorbent. The outcomes showed that this sorbent could quantitatively extract medications with the right amount of sensitivity, accuracy, and precision from water and wastewater samples(19).

Conclusion

In conclusion, we have made a system for the Dakin oxidation step of catechol synthesis that is aerobic and gentle. In this work, we investigated the production of hydrogen peroxide for the Dakin oxidation of various organic compounds using natural feedstocks such as WEWSA and WECMA. Our approach adapts to various groups that provide and accept electrons at the ortho-, meta-, and para-positions on the aromatic ring. It's remarkable that the catalytic system operates without the need for activation or the presence of a toxic ligand, additive, or promoter. So far, the developed catalyst system is the most effective and environmentally friendly way to enable Dakin to oxidize in air at room temperature. Three trials were conducted to evaluate the effect of time and 30% H_2O_2 on the conversion of salicylaldehyde into catechol at room temperature using WEWSA. As the third trial, 2 equivalents of H_2O_2 were used, and the reaction was carried out for 1 h, resulting in a yield of 70%. Similarly, how reaction time and 30% H_2O_2 affect the conversion of salicylaldehyde into catechol at room temperature (RT) using WECMA. In the case of the third trial, 2 equivalents of H_2O_2 were used, and the reaction was carried out for 1 h, resulting in a yield of 60%. This evidence indicates that as the concentration of H_2O_2 increases and the reaction time decreases, the yield of the reaction also increases. The information from the two conditions above suggests that changes in the catalytic conditions have minimal effect on product formation and yield. It also provides the synthetic community safe and effective oxidants. The oxidation protocol has the potential to save money and benefit the environment in both laboratory and industrial settings in the near future. These advantages make H_2O_2-WEWSA and WECMA strong options, providing a clean and simple choice compared to other major industrial reactions.

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