# Ultraviolet Radiation for Phenol Removal from Aqueous Solutions by Copper Oxide Nanoparticles in Advanced Oxidation Process

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**ABSTRACT:** *This study shows the efficient investigated processes of the Advanced Oxidation Process (AOP) (batch and continuous), which has been used for the degradation of the phenol in aqueous solutions by (UV/CuO nanoparticles). The effects of CuO nanoparticles' initial concentration, pH, and irradiation period were investigated in a batch system. The following ideal settings resulted in the maximum removal rate of Phenol from the stock solution (99%): In batch tests, 55 mg/L from CuO nanoparticles as a stimulant, irradiation time 120 min of and at pH 6. The effects of flow rate, number of UV lamps, and reaction time were investigated in a continuous system; the results showed that the efficiency of decomposition decreases as the flow rate of solution in the reactor increases; the maximum removal efficiency of the process (UV/CuO nanoparticles) was 90 % at 20 mL/min and 120 minutes of irradiation time. Copper oxide nanoparticles are useful in general because of their strong Phenol adsorption capability in the presence of UV, as well as the fact that they reduce the amount of Phenol in the stock solution.*


**KEYWORDS:** *Advanced Oxidation Process (AOPs); CuO nanoparticles; Phenol removal; Ultraviolet radiation.*

## INTRODUCTION

Phenolic chemicals have a wide range of industrial and everyday applications. They have a high water solubility and are very stable in the environment. Such chemicals, as well as other organic/inorganic substances and heavy metals, can pose major health risks to aquatic life. In terms of skin contact (irritant), ocular contact (irritant), ingestion, and inhalation, phenolic chemicals are extremely dangerous (lung irritant). Many phenolic compounds have potential applications as antioxidants, solvents or chemical intermediates, fire retardant additives in coatings and epoxy systems, plasticizers, polymerization, and so on. Environmental scientists have been studying the removal of phenolic compounds (and, in general, all organic/inorganic contaminants) because of their negative consequences[1-2].

Friedlieb Ferdinand Runge, a German chemist, was the first to extract phenol from coal tar in 1834. It has a benzene ring structure and is classified as an aromatic chemical. When placed under pressure at room temperature, it forms a hygroscopic crystalline solid. Pure phenol is a white solid, but when impurities are present, it becomes colored[3]. It dissolves in hydrocarbons like

benzene and polar solvents like ethyl alcohol and ether. In water, it behaves as a weak acid that can only partially dissociate. Coatings, rubber, and various types of plastic can be damaged by liquid phenol. Aluminum, lead, zinc, and magnesium may be destroyed by hot liquid phenol[4].

Currently, the separation and destruction approach can be used to remove phenol. Steam distillation[5], liquid-liquid extraction with various solvents[6], adsorption [7-8], membrane pervaporation[9], ion exchange[10], sedimentation [11], chemical oxidation[12], reverse osmosis and membrane-based solvent extraction are among the technologies for removing phenol from aqueous solutions and used in the separation procedure [13-14]. Total oxidation of phenol in water, wet oxidation with chemical oxidants, electrochemical oxidation, photocatalytic oxidation [14], supercritical water gasification, phenol degradation by electron discharges, biochemical degradation, the combination of degradation methods, and other techniques for providing energy for phenol destruction are all examples of phenol destruction methods. Among the many water treatment processes, adsorption is a quick, cost-effective, and universal procedure[15]. A batch procedure and a column operation are used in the adsorption technology for water purification[16-17].

Advanced Oxidation Processes (AOPs) are one of the most successful methods for degrading refractory substances. AOPs are processes that produce active hydroxyl radicals (OH) as a powerful oxidant for the breakdown and destruction of harmful materials using a variety of methods. The majority of AOPs are based on hydroxyl radicals because of their high oxidation capability (2.8 V). Ultraviolet radiation and oxidants such as $H_2O_2$[18], $O_3$[19], and $TiO_2$ are some of the most effective means of advanced oxidation[20]. Fenton[21], photo-Fenton[22], $UV/TiO_2$, and $UV/H_2O_2/O_3$[23] have all been employed in the removal of organic materials in recent years. Photocatalytic degradation occurs when pollutants are decomposed in the presence of metallic oxide particles such as ZnO[24] and $TiO_2$[25]. Titanium dioxide is a metallic oxide that is well known for degrading organic materials and is generally inexpensive, nontoxic, and water insoluble[4].

Among Advanced Oxidation Processes (AOPs), semiconductor based heterogeneous photocatalysis has counted the most famous AOP method for destroying organic pollutants. Final products of such process are carbon dioxide and water[26]. In this method, when photons of energy with higher or equal to the band-gap energy of the used semiconductor are absorbed, electrons from the valence band ($V_b$) are excited to its conduction band ($C_b$). This process generates the holes in $V_b$ and electrons in $C_b$ (e/h pairs). The resulted e/h pairs can react with dissolved oxygen and water molecules (or OH anions) and produce $O_2^{\bullet-}$ and $^{\bullet}OH$ radicals, respectively. These powerful non-selective oxidants can degrade different organic pollutants in aqueous media. Copper oxide (CuO), a semiconductor with an energy band gap of 1.2 to 1.5 eV and the ability to absorb UV/visible photons, was used in this study. $Cu^+/Cu^{2+}$ interactions have long been known to produce the oxidative alteration of numerous organic molecules. Cu can engage in redox reactions with both inorganic and organic molecules due to its unique interaction with the spin-restricted $O_2$ [27].

The studying goal is investing in the Copper Oxide Nanoparticles by Advanced Oxidation Process (AOP) for Phenol removal from aqueous solutions using AOP (UV/CuO nanoparticles) application.

## EXPERIMENTAL SECTION
### Nanoparticles Fabrication

CuOx nanoparticles are being more widely used as potential catalysts due to their nontoxicity, ease of manufacture, earth-richness compared to other materials, cost-effectiveness, flexible shape, high thermal and photochemical stability, and high-temperature super conductivity[28-29]. CuO is more appealing in practical applications than Cu2O because of its better stability[30].

MERCK made a blue tint by mixing 0.2 M copper acetate [$Cu(CH_3COO)_2$] in 100 ml distilled water and swirling it constantly on a magnetic stirrer at 85 °C. The pH was adjusted with dropwise injections of NaOH (98 %, Sigma–Aldrich) until it reached 12. After 6 hours of stirring at 85°C, black copper oxide nanoparticles were formed. The generated copper oxide nanoparticles were cooled at ambient temperature and rinsed continuously with distilled water until pH 7.0 was obtained in a centrifuge (REMI R-24, India) at 15000 rpm for 15 min. Finally, these nanoparticles were washed in 100% ethanol to remove impurities from their surfaces, dried in an 80°C hot air oven for 24 hours, and kept in glass containers for future use[31].

$$Cu(CH_3COO)_2 + 2NaOH \rightarrow \qquad (1)$$
$$Cu(OH)_2 + \downarrow 2CH3COONa \qquad [31]$$
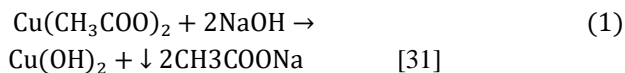
Calcination CuO (Black Nanoparticles)

SEM, XRD and FT-IR elemental mapping were used to analyze the produced CuO nanoparticles manufactured to determine the mechanism involved in the removal of Phenol [32-33].
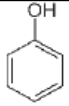
### Chemicals

Sigma Aldrich (M) Sdn. Bhd. sells phenol powder, which is kept in a refrigerator at a temperature of 2-8°C. To make the phenolic solution, it is dissolved in distilled water. Table 1 shows the chemical and physical properties of phenol. 0.1g of phenol powder is mixed with 1L of distilled water to make a 100 mg/L phenolic stock solution[7]. The phenol powder and distilled water mixture are then swirled with a magnetic stirrer to ensure that the phenol powder is completely dissolved. This stock solution is made in a room-temperature environment. The calibration curve must be established using five different concentrations of phenolic solution. Without additional purification, NaCl (99.5 %, Sinopharm) and $HNO_3$ (68 %, Fisher Chemicals) were utilized as received. The test solutions were made by dissolving the compounds in Milli-Q water purification system-generated ultra-pure water.

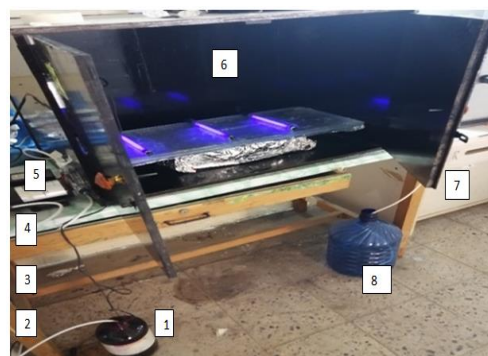### Batch Experiments for UV/CuO nanoparticles:

The batch process has been investigated in a beaker, magnetic stirrer was used for mixing the solution. Photocatalytic tests were conducted in a 250-ml glass beaker utilized for (AOPs). Glass beaker(batch reactor), 3 UV lights type(TL 8W BLB, Philips, Poland) has been put from the top, each one in 365 nm wavelength and 8 watts. At room temperature, from 10 mg/L to 65 mg/L of Copper oxide nanoparticles were used. The pH was from 2 to 8, and the irradiation time is somewhere between (30 -150) min. 150 mL from phenol stock solution put in the glass beaker, adjusted the pH then added CuO nanoparticles. The pH meter in type (Model: BP3001, Singapore) has been used for controlling the solution pH. At the beginning of the experiment, turn on the Ultraviolet lights and the magnetic stirrer at 250 rpm to guarantee homogeneous mixing to the mixture. The sample was finely obtained and evaluated using an Ultraviolet spectrophotometer at 291nm after the experiment was completed, as shown in Fig. 1.A.

*Table 1: Chemical and physical properties of phenol.*

| Formula | $C_6H_5OH$ |
|---|---|
| Molecular weight (g/mol) | 94.11 |
| $T_{melt}$ (°C) | 40.9 |
| $T_{eb}$ (°C) | 181.75 |
| Water solubility (r.t.) | 9.3 $g_{phenol}$ / 100 $mL_{water}$ |
| $\lambda_{max}$ | 9.89 |
| pKa | 270 nm |
| Flammability limits in air (vol%) | 1.7 (lower); 8.6 (higher) |



(a)



(b)

*Fig.1: A) Batch Process of Photocatalytic Oxidation. B) Continues process of Photocatalytic Oxidation.*

### Continuous Experiments for UV / CuO nanoparticles

As shown in Fig. 1.B. The box covering the reactor (6) was manufactured entirely from wood in size(90x 70 x 70) $cm^3$ and was painted black on the interior to ensure that Ultraviolet light was not obstructing the experiment. The box that houses the continuous photocatalytic glass reactor.

The reactor is (70x30x1) cm³ in size and is made up of two layers of glass. The thickness of top layer glass (6mm), while the reflexive mirror was used for the bottom layer, depth between these layers is 4mm. The solution circulates between the higher and lower levels of the system, in a feed plastic container (1) with a capacity of 1 liter, the solution was made. Water pump (2) was used to feed the phenol stock solution to the reactor, and flow meter(5) was posed to control the Phenol stock solution flow rate entering the continuous process.

The needed amount of photocatalytic powder was dissolved in 1 liter of phenolic stock solution for photocatalytic oxidation of phenol. The solution had a phenol content of 100 mg/L. By adding diluted $H_2SO_4$ solution to the feed plastic container (1), the pH has been calibrated at 6, and the necessary amount of CuO nanoparticles (10 mg/L) has been added to the feed plastic container(1), for 15 min the valve(4) should be closed for allowing the mixture to spread and ensure consistent mixing. After turning on the pump(2), allow the solution to flow from the feed plastic container (1) into the glass reactor photo-catalytic (6). Continuous glass reactor (6) ultraviolet lamp source was turned on, the calibrated flow meter was used to measure and control flow rates to (20, 30, 40, and 50) mL/min , collecting samples for testing, a valve (7) was employed. All of the experiments were carried out at room temperature.

Utilizing efficiency equation for calculating the Phenol removal from the aqueous solutions, as in Equ. (2)[31]:

$$\text{Phenol Efficiency (\%)} = \frac{Ci - Ct}{Ci} \times 100 \qquad (2)$$

Where: $Ci$: untreated Phenol concentration; $Ct$: treated Phenol concentration.

## RESULTS AND DISCUSSION
### Characteristics of the copper oxide nanoparticles produced

X-ray diffraction (XRD) research was used to determine the crystal structure of the CuO nanoparticles produced. Fig. 1 shows the XRD pattern of CuO nanoparticles produced. The XRD patterns reveal that all of the diffraction peaks for CuO (JCPDS 45-0937) are in good agreement with the standard diffraction data, and no characteristic peaks for other oxides were found (such as $Cu_2O$ or $Cu_2O_3$). The average crystallite diameters (D) of the samples were also determined from the principal diffraction peaks using the Debye-Scherer Equ. (3) [31].
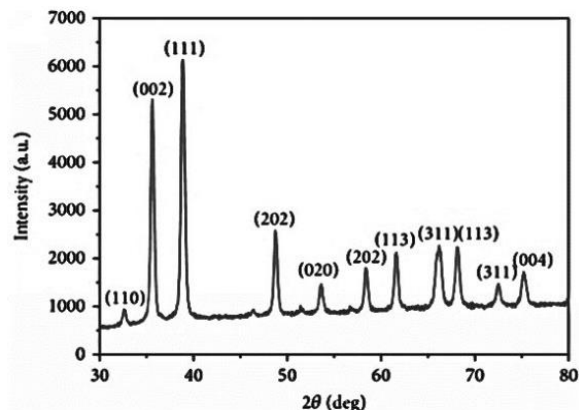


*Fig. 2: X-ray Diffraction. (XRD) of copper oxide (CuO) nanoparticles produced.*

$$D = \frac{0.94\,\lambda}{\beta\,\cos\theta} \qquad (3)$$

Where $\lambda$ is the wavelength of Cu Kα radiation, $\beta$ is the full width at half maximum height (FWHM) of the diffraction peak in radians, and $\theta$ is the Bragg angles of the main planes. The Debye-Scherer equation calculated the average crystallite size to be around 71 nm. Fig. 2. represented X-Ray Diffraction. (XRD) of copper oxide (CuO) nanoparticles produced.

SEM examination was used to analyze the surface morphology of the CuO nanoparticles produced. Fig. 3 shows SEM pictures of the CuO nanoparticles that were created. CuO nanoparticles have good homogeneity, spherical form, and adequate separation, as demonstrated, and their average particle size is around 50-70 nm. However, a few aggregates were found, which could be due to aggregation during the washing process. The particle size determined by the SEM investigation matches the XRD data quite well. The modest discrepancy between XRD's global picture and SEM analyses' local features can be explained in terms of the global picture. The Scanning Electron Microscope (SEM) pictures of CuO nanoparticles produced are shown in Fig. 3.

The vibrational characteristics of CuO nanoparticles were investigated using FT-IR spectroscopy, and a spectrum is shown in Fig. 4. As can be seen, the absorption bands at 3420 cm⁻¹ and 1624 cm⁻¹ correspond to the OH stretching and HOH bending modes of adsorbed water, respectively [34], with three discrete absorption peaks corresponding to the copper (II)–oxygen bond vibrations around 588 cm⁻¹, 534 cm⁻¹, and 435 cm⁻¹. The presence
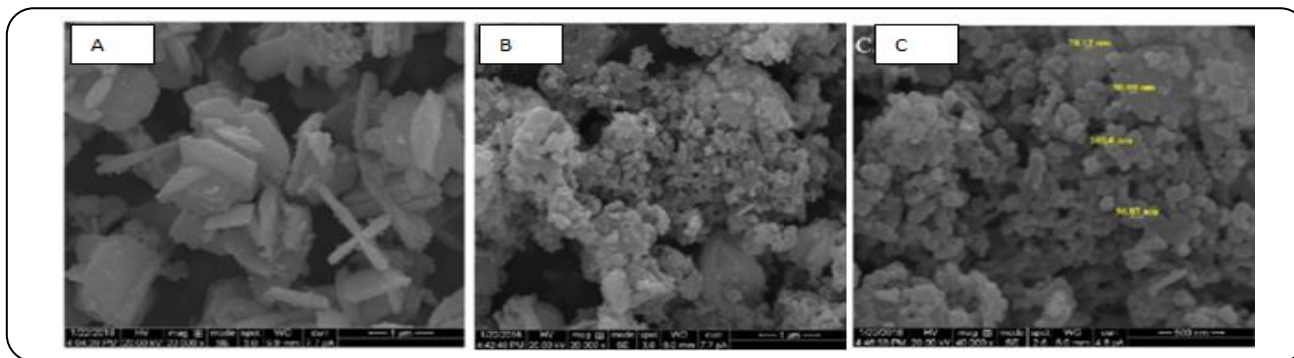
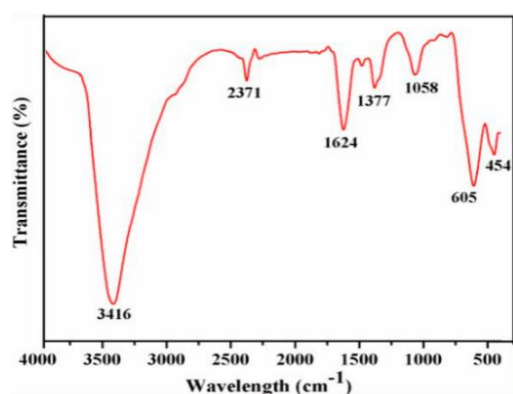*Fig. 3: Scanning Electron Microscope (SEM) images of CuO nanoparticles produced.*



*Fig. 4: Fourier Transform Infrared Spectroscopy (FT-IR) spectra of CuO nanoparticles produced.*



*Fig. 5: CuO nanoparticles dosage effects on Phenol removal at (120 min radiation time, pH 6 and 3 UV lamps).*

of ambient $CO_2$ causes a small dip in the spectra near 2363 $cm^{-1}$. Fig. 4 shows an FT-IR spectrum that is quite similar to CuO spectra previously published in the literature.

### *Photo-catalyst batch experiments*

*CuO Nanoparticles dosage effect on phenol removal*

Generally, for the pollutant degradation effects, it should recognize the photocatalytic dosage based on the interactivity between organic pollutants concentration into solution and the active sites quantity inside element; as a result, the performance of catalyst improved as the active sites quantity was increased. Different concentrations of CuO nanoparticles effects onto Phenol stock solution photochemical degradation (10-65) gm/L for (UV/CuO nanoparticles) process was investigated. Fig. 5 depicts the efficiency findings. Copper oxide nanoparticles concentrations were used from 10 gm/L to 65 gm/L to enhance the effectiveness of phenol removal, at 10 gm/L the Phenol removal 75%, and at 55 gm/L the Phenol removal 99%, when the radiation duration (90 min) and pH 6. When CuO
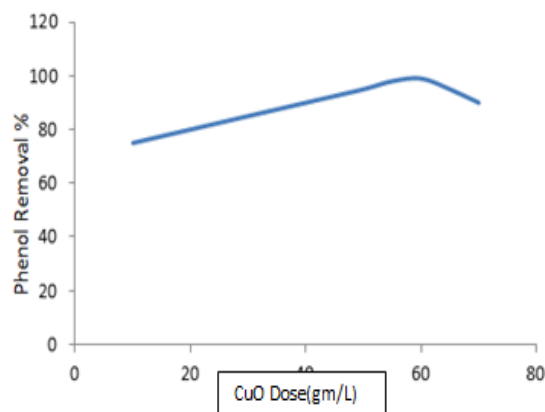
nanoparticles concentration was increased to 60 gm/L and 65 gm/L, the Phenol removal efficiency reduced to 95% and 90%, respectively).

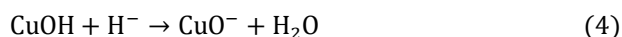### *Ultraviolet radiation time exposure effect on phenol removal*

The effect of the time it takes for UV radiation to break down the organic contaminants in Phenol stock solution was investigated in a range of scenarios (15-150) min. Batch experiments were used to investigate the link between irradiation period and phenol elimination by photocatalysis. Fig. 2 shows that as irradiation time rose, the percentage of Phenol removed increased, with150 min, being the highest irradiation exposure time and 120 min being the perfect irradiation exposure period Fig. 6. Generally, the electric consumption is very little for the photocatalytic treatment, that's equal to 60% of the total operation cost, thus the organic compounds were un degradable and stay in the same chemical structure if the time of pretreatment is very short.
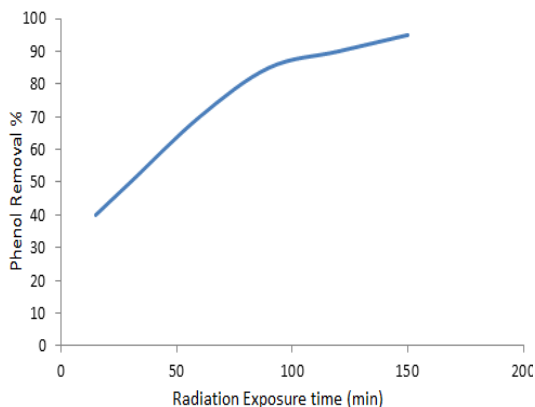
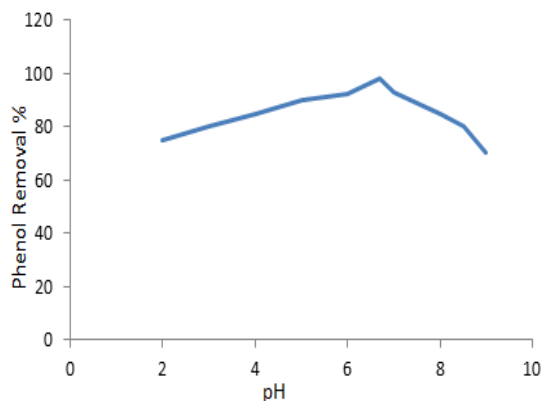**Fig. 6: UV radiation exposure time effects on Phenol removal % at(25gm/L CuO,25°C and pH 6).**



**Fig. 7: pH value Effects on Phenol Removal % at (55gm/L CuO, 120 min radiation time and 25 °C).**

*Effects of pH on phenol removal*

Because it affects the surface charge of the photocatalyst and the generation of hydroxyl radicals between hydroxyl ions and positive holes on the surface of CuO, the pH value of the solution could be a critical factor in the photocatalytic activity. CuO nanoparticles generated had a point of zero charges around pH 6. As a result, the CuO nanoparticles generated on the surface, for example, may suffer variations in charges as the pH of the solution varies:

$$CuOH + H^+ \rightarrow CuOH_2^+ \qquad (3)$$

$$CuOH + H^- \rightarrow CuO^- + H_2O \qquad (4)$$

where CuOH denotes the 'copperal' group on the surface. The efficiency of hydroxyl radical production may be affected by these modifications. The pH of the solution, on the other hand, can affect the surface charge of the catalyst as well as the protonation and deprotonation of the phenol molecules in the solution, resulting in a change in photocatalytic degradation kinetics and, as a result, a reduction in photocatalytic phenol degradation efficiency [32-33].

Solution pH influences the free radicals synthesis ratio formed during the photocatalytic treatment, which is a critically important component in treating Phenol stock solution. The influence of pH at various values on Phenol removal effectiveness in photocatalytic processes was explored utilizing (CuO nanoparticles = 55 gm/L; radiation period = 120 min) with varying pH to reach the greatest efficiency of Phenol removal. Fig. 3 shows that the greatest Phenol removal efficiency was (98%) when

pH was equal to 6 and that it steadily decreased to the minimum removal efficiency (80%) when pH was equal to 8 as shown in Fig. 7. As a result, pH lowering causes active area improvement for the catalytic agent, and for varied pH, the effective wide range can be a surface charge of catalyst, the removal efficiency has been affected by the particles of adsorbent that contact into the surface of the adsorbent.

***Experiments of Continuous Process***

This section has been investigated the Phenol stock solution flow rate, the number of Ultraviolet lamps that influence the reaction, and the Phenol elimination efficiency that affects the rate of reaction. The variable factors for estimating the continuous process were different from the others. Under various settings, the Phenol removal efficiency from the Phenol stock solution has been studied.

*Ultraviolet-Light Intensity Effect*

Changes in the number of Ultraviolet lamps used, one lamp, two lamps and three lamps for photocatalysis continuous process, each lamp in 365 nm wavelength and 8 Watt were used to investigate the impact of the Ultraviolet light on phenol elimination efficiency in the catalyzed photocatalytic reactor. The technique was put to the test to see how many Ultraviolet lamps were needed to remove Phenol effectively. Initial Phenol content in Phenol stock solution was 53.56 mg/L, pH at 6 and flow rate is 20 mL/min , Copper oxide nanoparticles concentration is 55 mg/L and duration exposure 120 min at room temperature) were kept constant. The findings
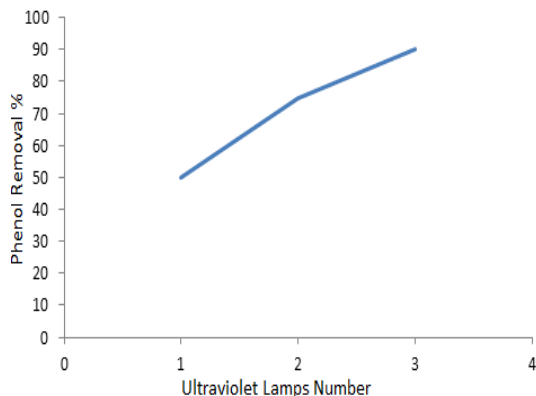
**Fig. 8: UV lamps number effects on Phenol removal at (55gm/L CuO, 120 min radiation time and 25 °C).**
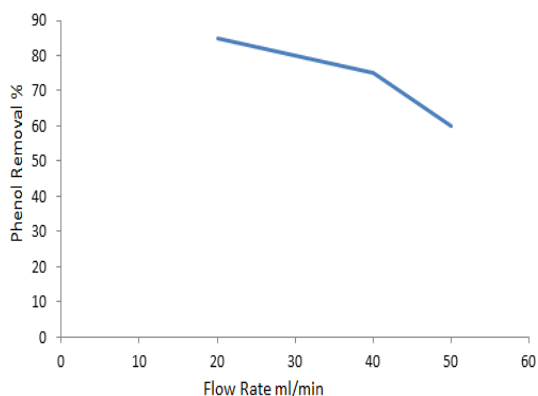


**Fig. 9: Flow Rate effects on Phenol removal (55 mg/L CuO, 3 UV lamps and 120 min radiation time).**
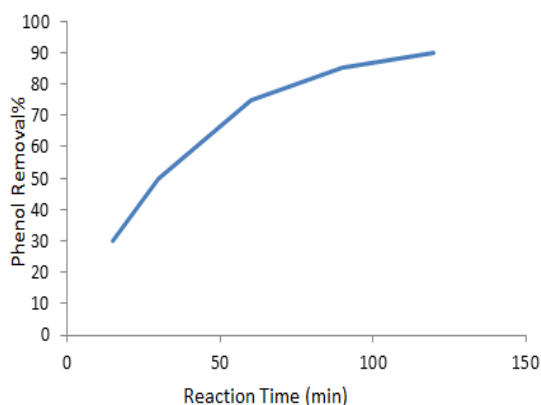


**Fig. 10. Reaction time effects on Phenol Removal (55 mg/l CuO =, 3 UV lamps and 25 °C).**

of the Phenol removal efficiency from Phenol stock solution increasing with the rise of the variable Ultraviolet lamps- A from 1 to 3 of the Ultraviolet lamp due to an increase in free radicals are depicted in Fig. 8.

*Flow Rate Effect on Removal Efficiency*

Flow rate is one of the substantial factors that must be taken into account in determining the proportion of Phenol recovered from the water Phenol stock solution that passes through the continuous photo-catalytic reactor made of glass. The Phenol removal efficiency was studied using ultraviolet light by changing the flow rate to (20, 30, 40, 50)mL/min , three Ultraviolet lights each one in 365 nm wavelength and 8 watts. Phenol stock solution high flow rate causes a negative effect on the Phenol removal quality, as shown in Fig. 9. Furthermore, the results revealed that as the flow rate of the Phenol stock solution is reduced, the efficiency of removing Phenol content improves, and vice versa. The elimination efficiency of the Phenol content is 85% at 20 mL/min  solution flow rate, and it rapidly decreases. It is clear from this that the pollutant particle residence time in the Phenol stock solution with CuO nanoparticles is a significant impact.

*Reaction Time Effect on Removal Efficiency:*

The reaction time of the Phenol stock solution is determined by the reactor's size and solution flow rate in the reactor. Following circumstances were used to investigate the influence of reaction time: 53.56 mg/L Phenol concentration in Phenol stock solution, pH at 6, three Ultraviolet lights, 55mg/L CuO nanoparticles concentration. The results show the rate of Phenol degradation was significant, which refers to owing to rising interaction between the Phenol stock solution and the surface of the photocatalyst Fig. 10. Phenol elimination effectiveness was 50% in the first 30 minutes. The Phenol elimination efficiency climbed to 75% after increasing the photo-reactor cumulative volume retention time to 60 min, and subsequently to 85% after 90 min. The Phenol degradation level remained impressively steady after increasing the radiation period to 120 min, with the effectiveness of Phenol elimination of 90%.

**CONCLUSIONS**

Using heterogeneous catalytic CuO nanoparticles for batch and continuous operations, the recovery of Phenol

content from the Phenol stock solution was investigated in this study. The organic contaminants in the Phenol stock solution were destroyed using a Copper oxide oxidant and ultraviolet light (UV/CuO nanoparticles). In a glass reactor, the Phenol content in a Phenol stock solution was decomposed. CuO nanoparticles are a very efficient oxidizing agent, a dependable and stable semiconductor for the removal of organic pollutants under UV irradiation, and the advanced oxidation process is an environmentally benign approach to eliminate the organic pollutants present in generated water. In a batch system, the ideal operating parameters were a very low catalyst 55 mg/L concentration of CuO nanoparticles and pH at 6. This resulted in a 99% removal efficiency of Phenol content. The impacts of flow rate, UV lamp number, and reaction time were tested in a continuous treatment system, and the maximum Phenol removal percentage was 90% at 120 min.

## Acknowledgments

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