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Rice husk-derived catalytic treatment of municipal wastewater: optimization using Box–Behnken design

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Abstract

Rice husk (RH) provides an abundant and sustainable source of silica, a crucial component in many catalysts. The most common uses of RH-derived catalysts are soil remediation, pollutant removal etc. The aim of present work is to optimize the catalytic treatment of municipal wastewater using Box–Behnken Design which is a type of experimental design commonly used in statistical modeling and optimization. Process variables such as pH, time (min) and catalyst dosage (mg/L) were optimized to get maximum Chemical Oxidation Demand (COD). Approximately 66% of COD removal was obtained at optimum operating conditions viz. pH: 5.9, time: 92 min, catalyst dosage: 246.27 mg/L. Software predicted maximum COD removal efficiency was found 75.6 % at optimal conditions.

Keywords: Municipal wastewater, Microalgae, Catalyst, Optimization, Central Composite Design

1. INTRODUCTION

After being contaminated by a number of applications, the community's water supply is mainly comprised of sewage and wastewater [1]. When it comes to generation wastewater can be liquid (or water) that includes waste from homes, schools, workplaces and factories, including groundwater, surface water, and rainwater [2]. "Sewage/Community/Municipal wastewater" often refers to the wastewater that is discharged from domestic areas like residences, institutions, and commercial establishments. It comprises carbon-based materials like human faeces, a piece of paper, vegetable matter, etc., making it organic compounds.

99.9% of it is liquid, and 0.1% is solid. Along with sewage from the nearby settlements, the area also contains industrial waste. Many industrial wastes are organic in nature, much like sewage, and are treated in various ways and/or by microbes.

Municipal wastewater treatment includes converting complex organic compounds into more manageable, odourless substances by physical, chemical, or biological means (biological treatment) [2]. Having untreated wastewater dumped in ground water, water from the surface bodies, or land has the following detrimental consequences on the environment:

- • The breakdown of the organic components in wastewater may result in significant amounts of pungent odours.
- • When without treatment wastewater (sewage) with a high amount of organic matter is dumped into a river or stream, the stream's dissolved oxygen supply is depleted in order to meet the wastewater's natural biological oxygen demand (BOD), that kills fish and has other negative effects [3].
- Additionally, wastewater may include nutrients that might encourage the development of aquatic organisms and algal blooms, resulting in the eutrophication of lakes, streams, and other water bodies.
- Untreated wastewater frequently contains a variety of hazardous substances and pathogenic bacteria that live in the human gastrointestinal system or may be found in particular industrial waste. These might pollute the area where the sewage is dumped or the water body. The aforementioned factors make wastewater treatment and disposal not just desirable but also required [3].

The elimination of toxic pollutants including ammonia, phenol, and hazardous metals both sewage and wastewater from industries has drawn considerable interest due to the risky nature of the growing number of health issues linked to environmental pollution. As it may offer matrix structure for nano composites, it has the potential to be employed as an inexpensive raw material to synthesise Nano composite catalyst to address environmental concerns. However, it's rare to find processes that utilise agricultural waste as a starting point to create advanced structures (Nano composites). However, researchers from all around the world are working to create a sustainable method for creating Nano composite catalysts from agricultural waste. Although manufactured ion exchange resin and carbon adsorption are efficient in treating wastewater, they are frequently unsuitable for the bulk removal of harmful pollutants due to their high cost. This prompted the study of alternative technologies that take into account affordability in addition to their capacity as adsorption materials. Therefore, the development of inexpensive sorbents is required for this purpose. One such strategy that has received considerable attention is the utilization of biological waste or industrial by-products. A variety of materials have been researched, including apple trash, tea leaves, moss, cotton husks, rice husks, cotton husks, and chitosan, to extract metal from wastewater. Due to its low energy, low cost, consumption, great efficiency, and ease of use, adsorption is a widely used approach for removing colours [4]. The adsorbent is a factor in one adsorption issue. In general, activated carbon is the preferred option, although the high price is a significant disadvantage. Utilising waste materials, particularly those from agro-industrial sources, which are inexpensive and widely available, is one approach to save expenses. Due to its large yearly output and presence of organic compounds, rice husk fits well within the notion of inexpensive adsorbents. However, due to its small surface area and inaccessible or non-functional locations, it performs poorly [5]. Alternative surface changes that enhancement the surface area and provide additional reacting sites can be done with the goal of improving adsorbent performance.

There have been several researches done on the purification of wastewater using rice husk. In India, rice husk, a by-product of rice, is widely accessible and in enormous supply. Typically, it is burned or disposed of as garbage. The primary components of the rice husk's cell walls include cellulose, silicon lignin, carbohydrates, and a significant amount of hydroxyl groups. Where the presence of their numerous functional groups is what gives the husk its exchange characteristics [6]. Due to its magnetic qualities and strong catalytic activity, nanoparticles of nickel are among the most often used as catalysts. Because of their tiny diameters and high surface-to-volume ratio, Ni Nanoparticles tend to self-aggregate, which lowers catalytic efficiency. Immobilising Ni Nanoparticles with high dispersion and massive loading volumes on the support may be the answer to stopping their self-aggregation. [7]. Biomass has received a lot of attention as a renewable source of energy in the creation of cutting-edge materials for a variety of applications because of their affordability, abundance, speedy regeneration, and environmental friendliness. Amazing amount of 2.9×10^7 tonnes of rice husks are produced as a by-product of the global rice milling industry each year. [8] The

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silica that rice receives from the soil as a live plant, in the form of silicic acid, builds up surrounding the cellulose micro-compartments [9]. As a result, rice husks are appropriate natural sources for creating high-value goods like catalyst supports or nanostructured silicate-based adsorbents. The present study emphasizes the optimization of process parameters during catalytic treatment of municipal wastewater using Box–Behnken Design.

2. MATERIALS AND METHODOLOGY

2.1. Materials

Rice husk obtained from local rice mill in Vadodara (10 -15% SiO2), Concentrated nitric acid (M.W. 63.01, 69-72%), Concentrated Sulphuric acid (M.W. 98.08, 98-99%), Sodium hydroxide (M.W. 40, 98%), Nickel sulphate hexahydrate (M.W. 262.85, 98%), distilled water were procured from local vendors of Vadodara. Municipal wastewater was collected from nearby area and initial COD was found 415 mg/L.

2.2 Methodology

2.2.1 Catalyst preparation

30 gram of dried rice husk (RH) was mixed with 500 mL of 1.0 M nitric acid and left for 24 hours at room temperature as shown in Fig. 1 (a). The rice husk was made free from NO_3 (-) by washing thoroughly with distilled water as shown in Fig. 1 (b). The acid treated rice husk was dried for 3 hours in an oven at 383K. The dried rice husk was then vigorously stirred in 500 mL of 1 M NaOH for 30 minutes and left at room temperature for 24 hours, than filtered to get sodium silicate (dark brown) solution as represent in Figure 1 (c).

Figure-1 (a)

Figure-1 (b)

Figure-1 (c)

Figure-1 (a) Dry RH added in HNO_3 solution (b) RH free from $NO3^-$ (c) RH in NAOH solution: Sodium Silicate solution

$NiSO_4 \cdot 6H_2O$ containing 10 wt.% Ni (2^+) (here 17gm) was completely dissolved in 130 mL of 3M Sulphuric acid as shown in Fig. 2 (a). This acid-containing metal solution was titrated into the prepared sodium silicate until pH 3 and the suspension obtained was left for ageing at room temperature for 48 hours. Soft gel was recovered from aged solution by using centrifuge device at 4000 RPM for 10 minutes as shown in Fig. 2 (b). The solid gel was oven dried at 383 K for 2 hours and ground finely. We obtain Nickel Nano-particles Silica Composite (Ni@SiO_2) Catalyst as shown in Fig. 2 (c).

Figure-2 (a)

Figure-2 (b)

Figure-2 (c)

Figure-2 (a) Solution of Sodium Silicate $NiSO_4$. $6H_2O$ and H_2SO_4 (b) Soft gel catalyst (c) Dried catalyst

2.2.2 Characterization of catalyst

BET is an important characterization technique to identify pore size, pore volume and surface area of particles. It was found that the BET surface area, pore volume and pore size of prepared catalyst was 56.5651 m^2/gm, 0.070621 cm^3/gm and 49.932 Å respectively. XRD analysis was also performed and observed crystalline structures with few phases of synthesized catalyst as shown in Figure 3.

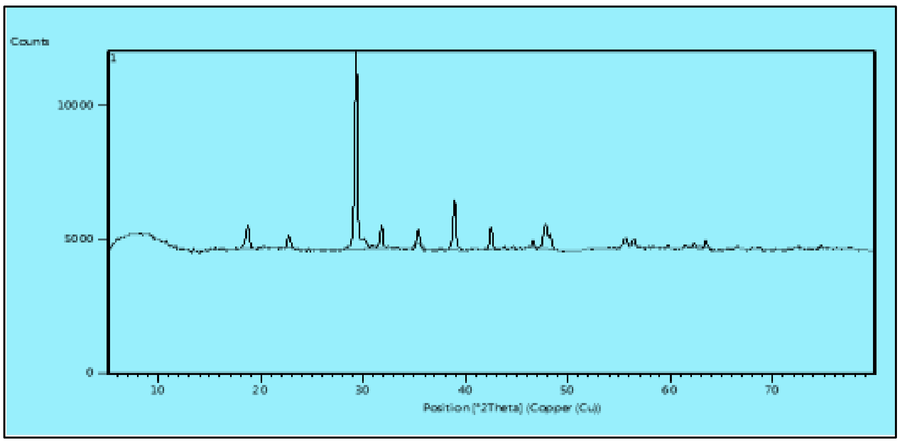

Figure-3: XRD plot of catalyst

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2.3 Optimization Study

Response Surface Methodology is an important process optimization tool mainly used to model and optimize complex systems by analyzing the relationship between the response variable (output) and the input variables (factors) of a process or experiment. In this study, box behnken design (BBD) under Response Surface Methodology was used to optimize process parameters such as pH: (3-9), time: (20-140 min) and catalyst dosage: (50-350 mg/L) during treatment sewage wastewater r as shown in Table 1. Twenty experimental sets were given by software as shown in Table 2. Trial runs were performed prior to finalizing the levels of variables

Table 1: Process parameters and their range

Design characteristics			
Levels	Parameter (Range)		
	pH	Time (min)	Catalyst dosage (mg/L)
-1	3	20	50
0	6	80	200
+1	9	140	350

Table 2: BBD predicted experimental sets

Run no.	variables		
	A (pH)	B (Time (min))	C (Catalyst dosage (mg/L))
1	3.00	20	200
2	9.00	20	200
3	3.00	140	200
4	9.00	140	200
5	3.00	80	50
6	9.00	80	50
7	3.00	80	350
8	9.00	80	350
9	6.00	20	50
10	6.00	140	50
11	6.00	20	350
12	6.00	140	350
13	6.00	80	200
14	6.00	80	200
15	6.00	80	200
16	6.00	80	200
17	6.00	80	200

3. RESULT AND DISCUSSIONS

3.1 EFFECT OF PROCESS PARAMETERS ON % COD REMOVAL DURING CATALYTIC TREATMENT

Study of the effect of process variables (pH, time and catalyst dosage) on COD removal was done at room temperature as shown in Fig. 4 (a, b & c). A fixed amount of hydrogen peroxide (20 mM) is used as an oxidant during each experimental run. In Fig. 4 (a), maximum removal of COD was achieved at optimum pH; 5.9. This might be due to the suppression of active sites on the catalyst surface or scavenging of hydroxyl radicals on catalyst surface in acidic medium and. Maximum removal efficiency is achieved at optimum catalyst dosage: 246.28 mg/L as shown in Fig 4(b). The less amount of catalyst dosage gives less number of active sites and insufficient space for reaction where excess dosage favors particles agglomeration in aqueous solution causes blockage of active sites lead to suppress hydroxyl radical's utilization and therefore removal decreased. The maximum COD removal was observed 66.64.5 % at optimum conditions as mentioned in Table 3.

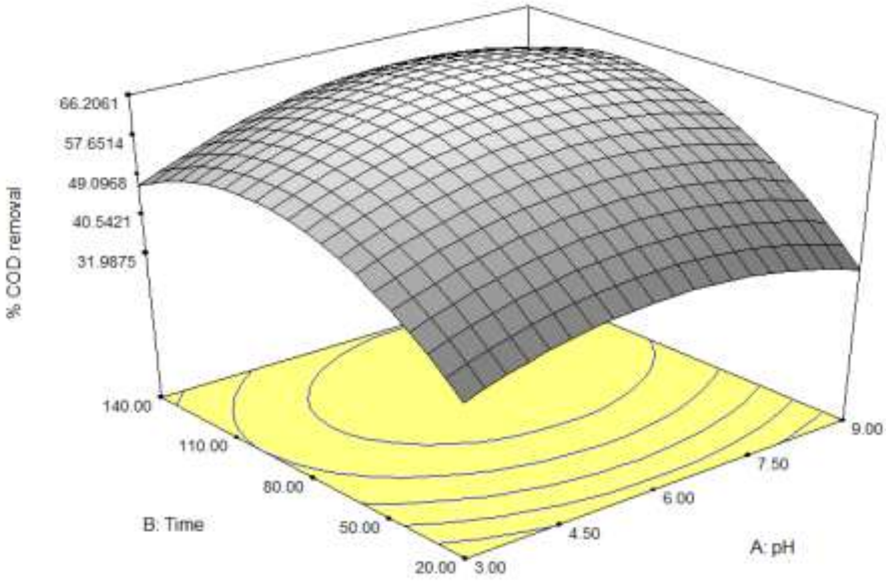

Figure-4 (a)

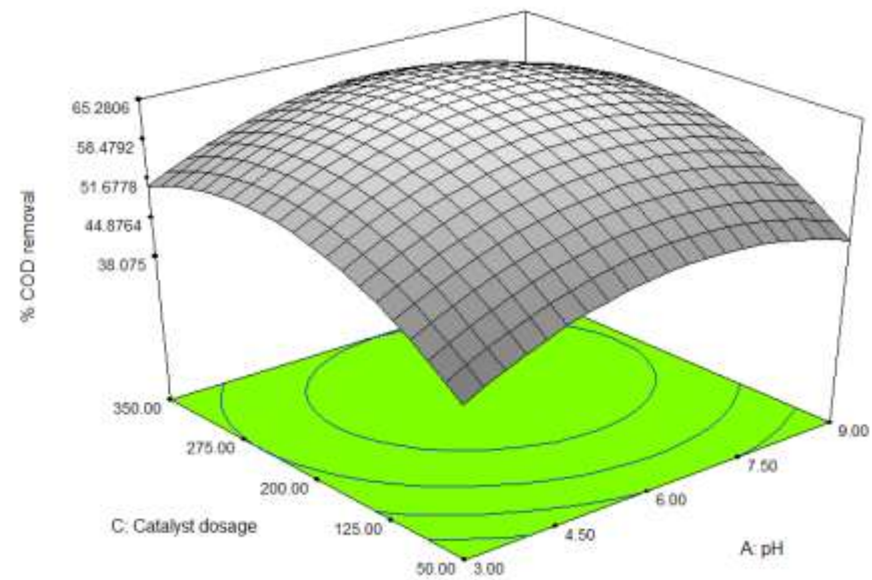

Figure-4 (b)

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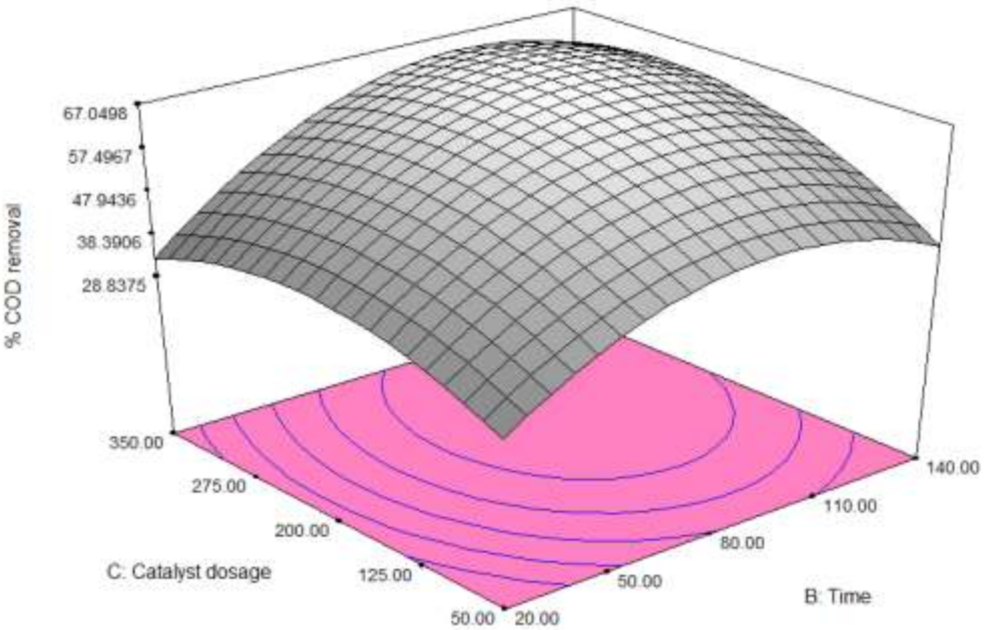

Figure-4 (c)

Figure-4 (a)(b)(c): Effect of pH, time and catalyst dosage on % COD removal

3.2. OPTIMIZATION:

Optimization of process parameters like pH, time, and catalyst dosage, were carried out by conducting set of experiments provided by box- behnken design as shown in Table 2. get Maximum COD removal at optimum conditions is shown in Table 3 and found proximity between model and test run predicted results.

Table. 3 Optimum conditions and removal efficiencies

pH	Time (min)	Catalyst dosage (mg/L)	% Removal of COD	
			CCD Pre.	Test run
5.9	92.02	246.28	66.64	63.4

5. CONCLUSIONS

Catalytic oxidation is a prominent and effective treatment technique in the present study. The effect of rice husk derived catalyst on treatment of municipal wastewater was investigated and found effective. Box-behnken design was used to optimize the process variables such as pH, catalyst dosage, and time during treatment and observed software predicted result which further analysed by test run shows successful design model. Approximately 66 % removal efficiency was predicted by the software which indicates satisfactory result and efficiency of model.

REFRENCES

[1] H. Zhou and D. W. Smith, Advanced Technologies in Water and Wastewater Treatment, J. Environ. Engg. Sci., 1, 247-264 (2002).

[2] N. P. Cheremisinff, Hank Book of Water and Wastewater Treatment Technologies, An Overview of Water and Water Treatments, Butterworth-Heinemann Publication, (2002) pp. 1-60.

[3] F. R. Spellman, Spellmann's Standard Handbook for Wastewater Operations, Vol. 1, 2 and 3, Lancaster PA, Technomic Publishers, (1999-2000) pp. 60-80.

[4] Dotto, G. L., Sellaoui, L., Lima, E. C. & Ben Lamine, A. Physicochemical and thermodynamic investigation of Ni (II) biosorption on various materials using the statistical physics modeling. Journal of Molecular Liquids 220 (1), 129–135

[5] Soltani, N., Bahrami, A. & González, L. A. Review on the physicochemical treatments of rice husk for production of advanced materials. Chemical Engineering Journal 264 (1), 899–935.

[6] E. Munaf & R. Zein (1997): The Use of Rice Husk for Removal of Toxic Metals from Waste Water, Environmental Technology, 18:3, 359-362.

[7] S. Zhang, S. Gai, F. He, Y. Dai, P. Gao, L. Li, Y. Chen, P. Yang, Uniform Ni/SiO2@Au magnetic hollow microspheres: rational design and excellent catalytic performance in 4-nitrophenol reduction, Nanoscale, 6 (2014) 7025-7032

[8] P. Chen, L.-K. Wang, G. Wang, M.-R. Gao, J. Ge, W.-J. Yuan, Y.-H. Shen, A.-J. Xie, S.-H. Yu, Nitrogen-doped nanoporous carbon nanosheets derived from plant biomass: an efficient catalyst for oxygen reduction reaction, Energy Environ. Sci., 7 (2014) 4095-4103.

[9] N. Liu, K. Huo, M.T. McDowell, J. Zhao, Y. Cui, Rice husks as a sustainable source of nanostructured silicon for high performance Li-ion battery anodes, Sci. Rep., 3 (2013) 1919-1925.