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Taguchi's Optimization Technique for Phenol Adsorption using Cotton Stalk Activated Carbon

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***Abstract*— This work demonstrates the utilization of the method of design of experiment as conceptualized by Taguchi to maximize the process variables for the adsorption of phenol from hydrous solution using the adsorbent of activated carbon prepared from cotton stalk (AC-CSW). Results obtained presented the influence of multiple variables such as the AC- CSW-dose (m), phenol-concentration (C_0), ambient-temperature (T), and the contact-time (t) on the response variable or character, adsorption capacity (q_e) at three levels using L_9 orthogonal array (OA) array, which in turn was examined using analysis of variance (ANOVA) on primary datasetset and Sound /Noice (S/N) ratio data using the 'larger is better' characteristic. Analysis of the ANOVA result obtained reveals that factor B (C_0) and A (m) are the most significant factors with a total of 45.21% and 47.84% contribution, respectively. The D (t) and C (T) factors are the least significant factors with a total of 4.18% and 2.77% contribution, respectively. The percent removal of phenol was noted to be 91.53% with an maximum uptake of 68.65 mg/g.**

***Index Terms*— Adsorption, ANOVA, Phenol, orthogonal array and Taguchi.**

I. INTRODUCTION

Scarcity of water and pollution is a critical environmental concern for nations globally [1]. The rapid growth in industrial civilization and urbanization has aggravated environmental contamination; among others, phenol and its derivatives have become some of the dangerous pollutants that need immediate deletion from water and wastewater streams [2][3]. Phenol (C_6H_5OH) is one such aromatic compound with high aquatic solubility and significant toxicity [4]. Acute exposure to it causes severe skin irritation, ocular damage, and respiratory distress, while chronic exposure causes renal dysfunction, hepatic damage, and even has the potential for carcinogenic effects [5]. This originates from various industrial sectors involving petroleum refineries, pharmaceutical manufacturing, pesticide production, leather tanneries, and paper mills [6]. Due to its aquatic toxicity and bioaccumulation potential, the United-State-Environmental-Protection-Agency (U-S-E-P-A) has classified phenol as a major contaminant needing effective management [7]. Traditional treatment methods like chemical oxidation and biodegradation exhibit low efficiency and require longer treatment periods [8][9].

The advanced methods using advanced oxidations, membrane filtration, and photocatalytic degradation present high efficiency, as well as demanding costs [10][11] Adsorption presents itself as the most feasible method due

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to the high removal efficiencies (90-99%), low operational cost, fast treatment kinetics, and scalability of the method at different production scales [12]. Though agri-waste based adsorbents have been demonstrated to be effective in phenol removal out of wastewater [13]. Activated carbon derived out of cotton stalk (AC-CSW), an agri-waste found in abundance in countries like India, has been scarcely investigated for phenol removal [14]. This particular problem presents an immense opportunity in making advancements in the field of sustainable water treatment available on an economic footing [15][16]. The current study aims to optimize the sorption of phenol on AC-CSW using Taguchi's robust design approach, needing only 9 experiments as opposed to 81, representing an 88.9% reduction in experiments required, preserving 95% of the required information using the concept of an orthogonal array [17][18]. The current study applies analysis of variance (ANOVA) and calculation of Signal/Noise (S/N) ratio to assign numerical values to factors, making an objective determination of their relative importance. This research aims to make an immense contribution in making progressive advancements in sustainable water treatment technology, making economic and environmentally sound technology accessible to Small and Medium Scale Businesses [19].

II. Materials and Methods

The cotton stalk, sourced from Nagpur, Maharashtra, India, was washed, naturally dried for 48 hours, and thermally dried at 105°C for 6 hours. The dried plant was processed into 0.5–2 mm-sized particles and treated with chemical activation by suspending in 1:0.5(g:mL) orthophosphoric acid (H_3PO_4) at 1g of adsorbent with 0.5ml of H_3PO_4 for 24 hours at ambient temperature. The treated sample was further processed by carbonization at a temperature range of 350±10°C in a muffle furnace for 1 hour, washed thoroughly by 2 M ammonia solution and distilled water until the pH is neutral. The prepared activated carbon derived from cotton stalk (AC-CSW) samples were dried at 120°C for 6 hours and analyzed by Scanning-Electron-Micrographs, X-Rays Diffraction, and Energy Dispersive Analysis of X-Rays.

A. Adsorbent

Phenol (C_6H_5OH) was obtained of merck grade from standard scientific cooperation Nagpur. 500 mg/L stock of phenol was prepared by dissolving 0.5 g of laboratory-grade phenol in doubly-distilled-water. The stock solution was kept at 4°C and then quantitatively diluted with double-distilled water to get required concentrations of phenol solution for conducting the experiment. The other chemicals used in this experiment, such as HCl, NaOH, KNO_3, and NaCl, obtained from Merck Specialities Pvt. Ltd., Mumbai, India, were of laboratory-grade quality and prepared using double-distilled water with a conductivity of not more than 2 μS/cm.

B. Taguchi's Design Methodology

Reference [20] describes Taguchi's experimental design methodology, which comprises the following systematic steps for process optimization: (1) choosing the output variable to optimize, (2) choosing the input variables that influence the response, called control factors, considering appropriate experiment levels for every control factor, (3) choosing an orthogonal array based on the number and experiment levels, (4) arranging control factors in the orthogonal array to obtain the Design of Experiment (DOE), (5) performance of the experiments based on the DOE, (6) dataset examined using Analysis of Variance (ANOVA) and computation of Signal/Noise (S/N) ratio to infer significance factors for optimizing optimal conditions, and (7) performance of the confirmatory experiment based on the optimal conditions for validating the optimization output

C. Output Variable and Control Factors

The adsorption capacity (mg/g) was chosen as the response variable for optimization. The four control factors are AC- CSW-dose, phenol-concentration, ambient-temperature, and contact-time, each with three levels. As shown in Table I, each factor was evaluated at three experimental levels. The experimental design used is based on the orthogonal array designed by Taguchi, which requires only 9 trials to generate 81 experiments with about 95% of information on process details.

TABLE I. Determination of Factors and their Levels for Adsorption of Phenol Over AC-CSW

Factor	Parameter	Units	Level:1	Level:2	Level:3
A	AC-CSW-dose (m)	g/L	2	4	6
B	Phenol-concentration (Co)	mg/L	50	100	150
C	Ambient-temperature (T)	°C	10	20	30
D	Contact -time (t)	min	30	60	90

D. Taguchi's L_9 Orthogonal Array Design

Taguchi's L_9 (3^4) orthogonal array was selected based on E = 2k + 1 equation (where k = 4 factors, yielding E = 9 experiments). This design presented in table II reduces 3^4 = 81 possible experimental combinations to 9 strategically selected runs (88.9% reduction) while retaining ~95% of required information through balanced level combinations.

E Design-Experiment (D.E)

A comprehensive D.E was developed by incorporating the control factors and corresponding levels from Table I into the Taguchi L_9 orthogonal array structure

F. Experimental Programme and Analysis

Nine experimental runs (in triplicate) were conducted according to the D.E presented in Table III. Each run was performed in 100 mL flasks containing 50 mL of phenol solution of specified concentration. The AC-CSW-dose and phenol-concentration, ambient-temperature and contact-time were varied as per D.E specifications in Table III. Samples were agitated at 120 rpm. Residual phenol was measured using UV-VIS spectrophotometry at 270 nm, and adsorption capacity was calculated as:

$$qe = (C_0 - C_e) \times V / m. \qquad (1)$$

Where
q_e= AC-CSW adsorption equilibrium capacity in mg/g.
C_0 = Initial phenol-concentration in mg/L.
C_e = Equilibrium phenol-concentration in mg/L. V= Phenol-volume in L.
m = AC-CSW-mass in g.
Experimental dataset were analyzed using ANOVA and Signal/Noise (S/N) ratio analysis. The "Larger-the-Better" S/N criterion was employed to maximize phenol uptake [15]. Confidence intervals (CIPOP for population; CICE for confirmation experiments) at 95% confidence level were calculated using standard Taguchi formulas with and pooled error variance V_e. Confirmation experiments validated the Taguchi-predicted optimal conditions [20].

TABLE II. TAGUCHI'S L9 (3^4) ORTHOGONAL ARRAY FOR PHENOL ADSORPTION ONTO AC-CSW

Run	A (m)	B (CO)	C (T)	D (t)
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

TABLE III. DESIGN OF EXPERIMENT (DOE) FOR PHENOL ADSORPTION ONTO AC-CSW

Run	A (m)	B (CO)	C (T)	D (t)
1	2	50	10	30
2	2	100	20	60
3	2	150	30	90
4	4	50	20	90
5	4	100	30	30
6	4	150	10	60
7	6	50	30	60
8	6	100	10	90
9	6	150	20	30

III. RESULTS AND INTERPRETATION

A. Material analysis of AC-CSW

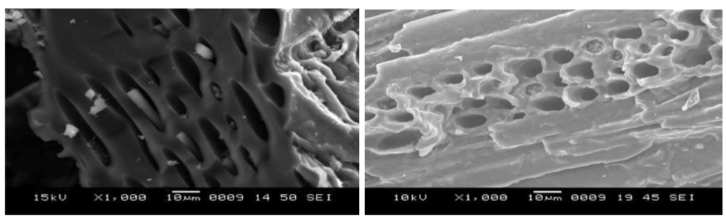
The surface area of activated carbon prepared from cotton stalk (AC-CSW), determined via N_2 gas adsorption using Brunauer-Emmett-Teller (BET) analysis, was 758.3 m^2/g before adsorption and 154.27 m^2/g after adsorption.There was a decrease of 604.03 m²/g in the surface area of AC-CSW following phenol adsorption. AC-CSW recorded 682.45 mg/g iodine number, indicating proper pore structure for adsorption. According to BIS 1350-I (1984), proximate analysis was conducted, with outcomes presented in Table IV. Proximate analysis revealed ash content of 13.24%, fixed carbon of 68.65%, moisture of 6.93%, and volatile matter of 11.18%, indicating effective drying and improved carbonization efficiency. The Figure1(a) and Figure1(b) are the Scanning-Electron-Micrographs (S.E.M.) of AC-CSW at 1000X magnification. The porous structure with irregular size micropores are seen on the surface without Phenol adsorption as depicted in Figure1(a) which are blocked and the surface is heavily loaded with Phenol adsorption as depicted in Figure1(b). The zero charge point of AC-CSW was determined to be 5.24 by the solid addition method [21].

B. L9 Orthogonal Array Experimental Findings

Experiments were conducted for phenol adsorption onto AC-CSW under conditions specified in TableV. Each experiment was conducted in triplicate. Average adsorption capacity values of qt were used for Taguchi's design analysis.

TABLE IV. PROXIMATE ANALYSIS OF AC-CSW

Sr. No.	Characteristics	AC-CSW (%)
1	Moisture content	6.93
2	Ash content	13.24
3	Volatile matter	11.18
4	Fixed carbon	68.65



(a) (b)

Figure 1. S.E.M picture of AC-CSW (a) without Phenol adsorption and (b) With Phenol adsorption onto surface

TABLE V. L9 ORTHOGONAL ARRAY EXPERIMENTAL DATASET: QT VALUES AND S/N RATIOS FOR AC-CSW PHENOL ADSORPTION

Run	Factor				Experimental result (qtmg/g)			Average qt	S/N Ratio (dB)
	A (m)	B(C_O)	C (T)	D (t)	R1	R2	R3		Average
1	2	50	10	30	24.15	24.08	24.12	24.12	27.65
2	2	100	20	60	47.32	47.89	47.25	47.49	33.50
3	2	150	30	90	68.43	68.91	68.65	68.65	36.72
4	4	50	20	90	13.28	13.28	13.21	13.26	22.44
5	4	100	30	30	25.18	25.07	25.24	25.16	28.02
6	4	150	10	60	39.45	39.42	39.44	39.44	32.21
7	6	50	30	60	8.91	8.93	8.93	8.92	19.01
8	6	100	10	90	17.63	17.63	17.56	17.61	24.93
9	6	150	20	30	26.37	26.37	26.37	26.37	28.42

C. Effect of Operational Parameters

The factors m, Co, T, and t significantly influence the phenol adsorption capacity (qt) of cotton stalk prepared activated carbon as shown in Table V. Factor A (m) demonstrate maximum effect at level 1, while factors B (Co), C (T), and D (t) show the highest influence at level 3 as shown in Figure 2 and Table VI. The differences between successive levels (L2–L1, L3–L2) indicate the relative impact of each parameter. Figure 3 and Table VII show the percentage influence of various parameters to the overall phenol adsorption on AC-CSW. Factor A (m, adsorbent dose) was found to be the most influential parameter, contributing 48.5% to the overall phenol removal. Factor B (C_0, initial phenol concentration) was the second most significant factor with a contribution of 44.5%. Factor D (t, contact time) showed a moderate effect with 4.0% contribution, whereas factor C (T, temperature) demonstrated the least influence on phenol adsorption, accounting for only 2.8% of the total variance. These results indicate that the process of adsorption for phenol on AC-CSW is primarily governed by the adsorbent dose and initial phenol concentration, while temperature plays a negligible role under the tested conditions. Based on Table VIII, the predicted value of Taguchi optimization on optimal adsorption capacity of 74.4 mg/g for the factor combination $A_1B_3C_3D_3$. Statistical validation at levels, establishing confidence intervals of 70.3-78.5 (population) and 72.2–76.6 confirmation experiment was 74.4 mg/g, which proves that the test result is included in the predicted confidence intervals and testing of the Taguchi model.

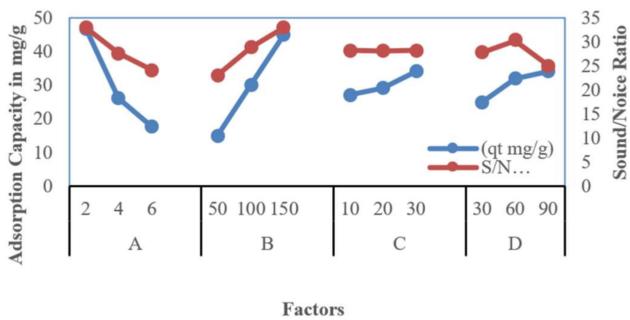


Figure 2. Impact of Process Variables on Phenol Adsorption by AC-CSW Across Factor Levels

TABLE VI. MEAN AND MAIN EFFECTS OF QT VALUES FOR AC-CSW: RAW AND S/N DATASET

Factor	Raw-dataset, Mean-value			Main-effects (Raw-dataset)		S/N Dataset, Mean-value			Main-effects (S/N Dataset)	
	L1	L2	L3	L2-L1	L3-L2	L1	L2	L3	L2-L1	L3-L2
A	46.70	26.03	17.62	-20.67	-8.41	32.62	27.55	24.12	-5.07	-3.43
B	15.45	30.23	44.48	+14.78	+14.25	23.03	28.81	32.50	+5.78	+3.69
C	27.15	29.05	34.10	+1.90	+5.05	28.03	28.17	28.14	+0.14	-0.03
D	25.19	32.07	33.05	+6.88	+0.98	27.99	28.25	28.10	+0.26	-0.15

TABLE VII. ANOVA FOR PHENOL ADSORPTION ON AC-CSW

Factors	ANOVA qt					Pooled-ANOVA qt				
	S	D. O. F	V	F	P	S	D. O. F	V	F	P
A	3600.00	2	1800.00	180.00	48.5	3600.00	2	1800.00	180.00	48.3
B	3300.00	2	1650.00	165.00	44.5	3300.00	2	1650.00	165.00	44.3
C	210.00	2	105.00	10.50	2.8	0.00	0	0.00	0.00	0.0
D	300.00	2	150.00	15.00	4.0	300.00	2	150.00	15.00	4.0
Error	200.00	20	10.00	1.00	3.2	210.00	22	9.55	1.00	3.4
Total	7610.00	26			100.0	7410.00	26			100.0

Note: S=sum of squares; D.O.F=degree of freedom; V=variance; F=variance ratio; and P=% influence

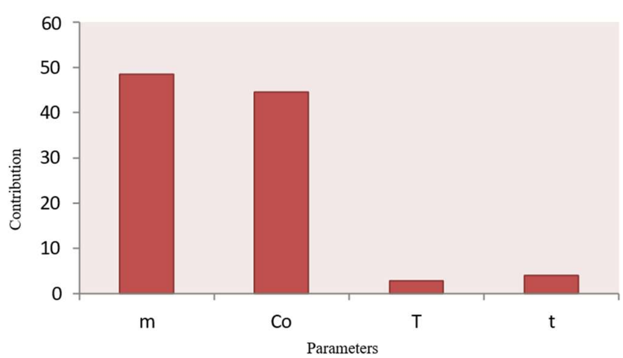


Figure 3. Parametric percent contribution for Phenol AC-CSW adsorption

TABLE VIII. CONFIRMATORY EXPERIMENTAL RESULTS

Sample	Optimizes Parameter	Predicted optimum(mg/g)	CI 95%	Confirmation Experimental Mean (mg/g)
AC-CSW	$A_1B_3C_3D_3$	74.4	CI_{POP}: $70.3<\mu_{AC\text{-}CSW}<78.5$	74.4
			CI_{CE}: $72.2<\mu_{AC\text{-}CSW}<76.6$	

IV. Conclusion

The Taguchi design of experiments achieved successful results for phenol adsorption optimization on cotton stalk-derived activated carbon (AC-CSW) which reached its maximum capacity of 74.4 mg/g at optimal conditions $A_1B_3C_3D_3$ (AC-CSW dose: 2 g/L, phenol concentration: 150 mg/L, temperature: 30°C, contact time: 90 min), which experimental tests verified at 74.4 mg/g between 95% confidence intervals (CIPOP: 70.3–78.5 mg/g; CICE: 72.2–76.6 mg/g). The analysis of variance showed that AC-CSW dosage (48.5%) and initial phenol concentration (44.5%) represented the main factors, but temperature had minimal effect, which demonstrated that adsorption happened mostly through mass transfer and concentration gradient processes. The L_9 orthogonal array allowed experimental trials to decrease from 81 to 9 runs, which maintained 95% process information. The method used agricultural waste as a cost-effective, eco-friendly solution for small- and medium-scale industries to implement scalable phenol removal.

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References

[1] S. L. Postel, G. C., Dailey and P. R. Ehrlich, "Rivers for life: Managing water for people and nature", Island Press, 2003. https://doi.org/10.5822/978-1-61091-029-7

[2] R. P. Schwarzenbach, T. Egli, T. B. Hofstetter, U.von Gunten, and B. Wehrli, "Global water pollution and human health", Annual Review of Environment and Resources, 35, 109–136. 2010. https://doi.org/10.1146/annurev-environ-031809-134701

[3] T. Gupta, A. Dharaskar, S. Gupta, and A. Jaiswal, "Sustainable Waste Management Through Development of Organic Composts Using Laboratory Synthesized Liquid Activators," International Journal of Environmental Research, 2025, 19(6):268-290. https://doi.org/10.1007/s41742-025-00932-2

[4] IARC (International Agency for Research on Cancer). Phenol and phenol derivatives. IARC Monographs on the Evaluation of Carcinogenic Risks to Humans, 100F, 345–356 (2012).

[5] NIH (National Institutes of Health). Phenol toxicity study. National Center for Biotechnology Information, PubChem Compound Summary, 2021. https://pubchem.ncbi.nlm.nih.gov/compound/996

[6] R. Ganesh, G. Balaji and K. Ranganathan, "Renibacterium salmoninarum removal from wastewater by biosorption using Sargassum sp. (brown algae)", Journal of Hazardous Materials, 133(1),61–67 2006. https://doi.org/10.1016/j.jhazmat.2005.09.045

[7] United States Environmental Protection Agency. Priority pollutant list.EPAOfficeofWater,Washington, DC, 2018. https://www.epa.gov/trinity/priority-pollutant-list

[8] K. P .Singh and A. Nayak, "Removal of phenol from wastewater using fly ash prepared from mineral coal", Journal of Hazardous Materials, 109(1-3), 2004. 73–81. https://doi.org/10.1016/j.jhazmat.2004.02.009

[9] T. Gupta, K. Ansari, D. Lataye, M. Kadu, A. Mohammad, N. Mubarak, R. Garg, and R. Karri, "Adsorption of indigo carmine dye by Acacia nilotica sawdust activated carbon in fixed bed column," Scientific Reports, 2022, 12: 15522. https://doi.org/10.1038/s41598-022-19595-6

[10] M. A. Oturan and J. J Aaron, "Advanced oxidation processes in water/wastewater treatment: Principles and applications", Critical Reviews in Environmental Science and Technology, 44(23), 2577–2641. 2014. https://doi.org/10.1080/10643389.2013.829765

[11] T. Gupta and D. Lataye, "Adsorption of indigo carmine dye onto Acacia nilotica (babool) sawdust activated carbon", ASCE Journal of Hazardous, Toxic, and Radioactive Waste, 2017, 21(4): 04017013. https://doi.org/10.1061/(ASCE)HZ.2153-5515.000036

[12] M. Ahmaruzzaman, "Adsorption of phenolic compounds on low-cost adsorbents: A review". Advances in Colloid and Interface Science, 143(1-2), 48–67,2008 . https://doi.org/10.1016/j.cis.2008.07.002

[13] P. T. Dhorabe, R. S Ingole and , D. H Lataye. "Adsorption of p-nitrophenol onto acacia glauca saw dust using Taguchi method". International Journal of Environmental Science and Development, 7(8), 605–609. 2016. https://doi.org/10.18178/ijesd.2016.7.8.865

[14] S. Dutta, B. C.Bhattacharyya and B. K. Mishra, "Removal of phenol from wastewater using charcoal from pyrolysis of agricultural waste", Bioresource Technology, 79(1), 15–20 2001 https://doi.org/10.1016/S0960-8524(01)00022-6

[15] V. C.Srivastava, M. M. Swamy, I. D.Mall, B. Prasad and I. M. Mishra, "Adsorptive removal of phenol by bagasse fly ash and activated carbon: Equilibrium, kinetics and thermodynamics", Colloids and Surfaces A: Physicochemical and Engineering Aspects, 272(1-2), 89–104 2007. https://doi.org/10.1016/j.colsurfa.2005.07.016

[16] T. Gupta and D. Lataye, "Removal of crystal violet and methylene blue dyes using activated carbon of Acacia nilotica sawdust", Indian Journal of Chemical Technology,2019,26: 52-68. https://nopr.niscpr.res.in/bitstream/123456789/46985/1

[17] J. Antony, “Design of experiments for engineers and scientists”, Butterworth-Heinemann, 2001. https://doi.org/10.1016/B978 075065214-8/50001-3
[18] T. Gupta and D. Lataye, “Adsorption of indigo carmine and methylene blue dye: Taguchi’s design of experiment to optimize removal efficiency”, Sadhana, 2018, 43(10): 1-13. https://doi.org/10.1007/s12046-018-0931-x
[19] United Nations, Sustainable Development Goals: Goal 6 - Clean water and sanitation. UN Sustainable Development Programme,2015 . https://sustainabledevelopment.un.org/sdg6
[20] M. B Silva, L. M. Carneiro, J. P. A.Silva , I. D.S Oliveira., H. J. I. Filho and C. R. O. Almeida , “An application of Taguchi method (Robust Design) to environmental engineering: Evaluating advanced oxidation process in Polyester-Resin wastewater treatment”, American J. of Analytical Chemistry, 5, 828-837, 2014.
[21] D. H. Lataye, I. M. Mishra and I. D.Mall , “Removal of pyridine from Aqueous Solution by Adsorption on Bagasse Fly Ash”, Ind. Eng. Chem. Re., 45(11), 3934–3943, 2006.