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Agricultural By-products: Optimizing Production of Activated Carbon using the Taguchi Method

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Abstract. This study optimizes the production of activated carbon (AC) from two distinct agricultural wastes, barley husk and maize cob. The process is refined using the Taguchi method with the goal of enhancing water treatment techniques. The study emphasizes how crucial it is to make the transition towards renewable and affordable raw materials for the manufacturing of AC given the predicted expansion of the worldwide demand for AC. The study assessed the impact of four crucial variables on the production and quality of AC using a L9 orthogonal array: phosphoric acid concentration, heating by microwave time, power from the microwave, and nitrogen flow rate. Findings indicated that the most important element for maximizing the production of AC from these agricultural leftovers was microwave power, along with the percentage of phosphoric acid and the time frame of the heating process.

Keyword-: Biomass, agricultural waste, activated carbon, Taguchi Method, orthogonal array.

1 Introduction

Anions, heavy metals, organic contaminants, and colorants have all been removed from water supplies using various techniques in the recent years. Water can be treated simply and efficiently with adsorption technology. AC (activated carbons) has been demonstrated to be a reliable adsorbent in several applications, such as wastewater treatment, water from the ground treatment, and removing organic molecules and mercury oxides from gas mixtures. In gas and liquid settings, its tiny pores, wide surface area, and ideal pore size distribution result in outstanding adsorptive properties. Its heat resistance, speedy adsorption rates, and low reactivity to acids and bases further enhance its remarkable adsorptive properties. Its

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manufacturing costs are a major obstacle to AC's extensive commercial acceptance. As low-cost starting materials for AC, agricultural waste and by-products such as rice husks, coconut shells, and palm oil fibres have been identified. Agriculture by-products are increasingly being researched for their potential to produce activated carbon (AC). The process offers two advantages at once: it produces an environmentally friendly by-product while ensuring agricultural residues are managed efficiently. In 2012, activated carbon (AC) accounted for $2007 million in revenue; by 2020, it is expected to reach $5305 million [3]. Approximately 13.3% CAGR (compound annual growth rate) is expected from 2014 to 2020 for the market expansion. Chemical properties of AC are determined by the surface groups and chemical bonds formed with heteroatoms. In addition to polarization intensity, hydrophobicity, acidity, and adsorption capacity, these surface groups determine polarization intensity, hydrophobicity, and acidity. A number of factors, such as biomass's physicochemical characteristics, oxygen content, electrical and catalytic characteristics, and the particular pollutants to be eliminated, determine the optimal temperature needed to activate AC [4].

It has drawn global interest to produce environmentally friendly, affordable activated carbon (AC) out of lignocellulose biomass as an answer to environmental waste management. There are usually two methods for fabricating AC from biomass [4,5]. Among these, chemical activation is frequently chosen because it requires lower temperatures, takes a shorter amount of time to carbonize, and consumes less energy. It is preferred to activate with phosphoric acid (H3PO4), but sulphuric acid (H2SO4), zinc chloride (ZnCl2), ferric chloride (FeCl3), sodium hydroxide (NaOH), potassium hydroxide (KOH), and potassium carbonate (K2CO3) are also popular chemistries [6]. The main reasons for its popularity in the recovery process are its ability to produce a greater yield of carbon and its small environmental impact. A number of techniques have been applied to eliminate the pollutants in water/ wastewater, like, ion exchange, adsorption, evaporation, reverse osmosis, and precipitation. Activated carbon adsorption is one of the most promising methods for removing the color, nitrate, and other contaminants in waters. Although the practice is quite effective, activated carbon is an expensive material to produce [7, 8]. Commercially, in the production of ac, carbon-rich materials are best preferred. These usually comprise wood, coconut shells, and coal. In addition to being expensive, these materials usually have to be imported, which increases the cost even more. Therefore, it is imperative to investigate affordable and easily available raw materials for the production of AC. This is extremely important for businesses that handle wastewater and purify water, especially in underdeveloped countries. [10, 9].

2 Taguchi Analysis

The Signal-to-Noise (S/N) ratio is used by the innovative Taguchi method to optimize processes. The optimization approach consists of two steps: determining control factors that lower variability and those that shift the mean toward the target while maintaining the S/N ratio. When an outcome or procedure is not impacted by outside variables like humidity, temperature, or weather, it is said to have a robust design. In order to reduce the susceptibility of a control characteristic to noise influences, the S/N ratio combines mean and variance. For optimization issues, S/N ratios come in three different varieties. For optimization issues, there are three different kinds of S/N ratios: nominal-the-better, larger-the-better, and smaller-the-better. Smaller-is-better works well for goals that have a target value of zero, little output or response, and non-negative data characteristics. The larger-the-better technique is appropriate for positive data characteristics and maximum output, but the nominal-the-better technique performs most effectively for values having either positive or negative traits. Sunflower seed extracts from plants, phosphoric acid, zinc chloride, and the Taguchi method can all be used to create high surface area activated carbon. For this carbon, high activation temperatures are advised. Researchers looked into how well banana trunk activated carbon removed nutrients from artificial greywater.

Table 1: Using Taguchi method analysis of activated carbon obtained from different agricultural products

Study	Material	Focus	Important Factors	Findings
[16]	Sunflower seed extracted meal	Activated carbon surface area	Activation temperature	Ideal activation temperature at 600°C, impregnation ratios for ZnCl2 and H3PO4
[17]	Banana trunks	Nutrient removal in greywater	Zinc chloride (ZnCl2)	ZnCl2 most important for nitrate removal
[17]	Banana trunks	Nutrient removal in greywater	ZnCl2, impregnation duration	ZnCl2 and impregnation duration for nitrate, ZnCl2 and activation time for phosphate
[18]	Rubber seed shells	Activated carbon manufacture	Activation temperature, chemical reagent concentration	Yield and amorphous percentage optimized
[19]	Coconut leaflets	Activated carbon production	H3PO4 and ZnCl2 step, KOH phases	Larger iodine number and surface area with specific activation method
[20]	Palm kernel shell	Methane adsorption	Activation temperature, impregnation ratio, activation duration	Highest methane uptake at 900°C, impregnation ratio 0.55, and duration 150 minutes
[21]	Schima wallichii biomass	Adsorption of Alizarin Red S dye	pH, adsorbent dose, dye concentration	Optimum adsorption at pH 3, 0.3 g dose for 200 mg L−1 concentration
[22]	Schima wallichii biomass	Adsorption of Alizarin Red S dye	Adsorption isotherm, thermodynamics	Endothermic and spontaneous adsorption process
[23]	Magnetic nanoparticles (Fe3O4) on AC	Cu(II) removal from aqueous solution	Activation parameters	Optimal properties for Cu(II) removal synthesized
[24]	Lignocellulosic residues	Dye adsorption	Specific surface area, micropores, mesoporosity	High surface area and porosity important for dye adsorption

The removal of phosphate and nitrate is influenced by variables such the impregnation ratio, activation period, and ZnCl2. Amorphous percentages and yields can reach ideal levels under certain conditions. In the study, the activation duration influenced the amorphous percentage; the activation temperature influenced the product yield. Chemically activating coconut leaflets using potassium hydroxide, zinc chloride, and phosphoric acid is studied in [19]. The best production method involved one step for H3PO4 and ZnCl2, two phases for KOH activation, and a larger iodine number and surface area. The final activated carbons' morphological, textural, and functional properties were examined. The purpose of the study in [20] was to use Taguchi orthogonal array design to optimize the synthesis of activated carbon from palm kernel shell for methane adsorption. The ideal parameters were an activation temperature of 900°C, an impregnation ratio of 0.55, and an activation duration of 150 minutes. In comparison to other settings, the largest methane uptake was observed by activated carbon manufactured under ideal conditions, which had the highest BET surface area of 1,548.0m2/g and the total pore volume of 1.0 cm3/g. The Freundlich isotherm was fitted by the maximum methane uptake under ideal circumstances. According to the work in [21], Alizarin Red S dye may be adsorbed using activated carbon made from Schima wallichii feedstock. Numerous techniques, such as the FT-IR, FE-SEM, and surface area of the BET analyzer, were utilized for evaluating the adsorbent. The process parameters were optimized using the Taguchi technique and ANOVA. The ideal conditions were discovered to be at pH 3 with a recommended dosage of adsorbent of 0.3 g and a dosage of dye of 200 mg L−1. After fitting the adsorption thermodynamic and kinetics to the experimental results, an optimal adsorption capacity of 91.695 mg g−1 was achieved by the adsorption isotherm, which obeyed the Langmuir model. Thermodynamics, analyses demonstrated the endothermic and instantaneous the adsorption of Alizarin Red S onto Schima wallichii carbon that is activated [22].

The effectiveness of the rejuvenated adsorbent was demonstrated by regeneration experiments up to the fourth regenerating cycle. In order to effectively remove Cu(II) from aqueous solution, magnetic nanoparticles (Fe3O4) were produced and adsorbed on activated carbon (AC) in the study [23]. Utilizing the Taguchi technique, activated carbons with the best qualities for eliminating food and textile colorants were created from various lignocellulosic wastes. Mesoporosity was critical to the absorption of these dyes, having the ideal carbons featured micropores and a large specific surface area [24]. Table 1 displays the utilizing Taguchi method's development of activated carbon derived from various sources of agriculture.

3 Design of experiments

The Taguchi method was utilized in this study to streamline the experimental process, which resulted in fewer experiments being required to determine optimal conditions. The effect of various control factors is evaluated independently using an orthogonal array in this robust statistical technique. A strategic selection of process variables was aimed at optimizing activated carbon (AC) yield. S/N ratios were calculated using the criterion "greater is better" in order to quantify the effectiveness of the experimental findings. Each control factor was assessed at three different levels as part of the L9 orthogonal array. In the study, factors such as the concentration of the activating agent (Factor A), the duration of radiation exposure (Factor B), and the intensity of microwave power (Factor C) were taken into account. With a flow control mechanism at the top of the microwave oven, nitrogen gas was fed directly into the system. Each factor was tested at different levels, which are detailed in Table 2.

Table 2: The level of each factor

Factors	Level 1	Level 2	Level 3
Phosphoric Acid (wt%/v)	30	60	90
Time (min)	2	4	6
Power (KW)	0.2	0.4	0.6
Nitrogen flow (cm3/min)	100	200	300

As part of the experimental design, noise factors are considered in the layout of the L9 orthogonal array. Taguchi's L9 orthogonal array technique for design of experiments is implemented in Minitab, a statistical software program the analyzes data with Taguchi's L9 orthogonal array technique.

Table 3: DOE Orthogonal array (L-9)

Phosphoric Acid (wt%/v)	Time (min)	Power (KW)	Nitrogen flow (cm3/min)	barley husk	corn cob
30	2	0.2	100	95.61	94.21
30	4	0.4	200	92.28	91.78
30	6	0.6	300	94.97	95.34
60	2	0.4	300	96.29	98.5
60	4	0.6	100	98.21	97
60	6	0.2	200	94.03	97.58
90	2	0.6	200	97.7	94.71
90	4	0.2	300	95.98	98.2
90	6	0.4	100	92.27	88.77

4 Discussion

The results obtained from the Taguchi analysis were crucial in identifying the most influential factors affecting the production of activated carbon. The analysis revealed that microwave power held the highest significance in influencing the activation process for both barley husk and corn cob, indicating its critical role in the efficiency of activated carbon production. Although the impact of phosphoric acid concentration and microwave heating time varied between the two agricultural wastes, they were also significant factors that required careful optimization. Nitrogen flow rate was identified as the least influential factor, implying that while it has an effect, it is not the primary driver of performance. The delta values and rankings provided by the response tables 3-4 for signal to noise ratios and means were instrumental in prioritizing the process parameters for optimization.

4.1 Taguchi Analysis: barley husk versus Phosphoric Acid (wt%/v), Time (min), Power (KW), Nitrogen flow (cm3/min)

Table 4: Analyze the response of signal-to-noise ratios

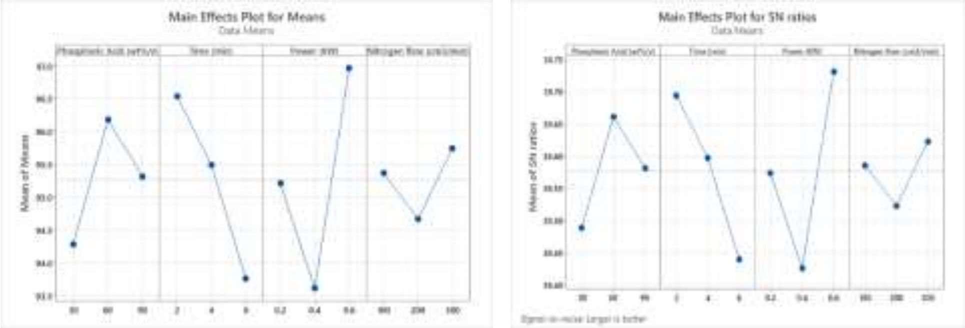
Level	Phosphoric Acid (wt%/v)	Time (min)	Power (KW)	Nitrogen flow (cm3/min)
1	39.49	39.69	39.57	39.58
2	39.66	39.60	39.42	39.52
3	39.58	39.44	39.73	39.62
Delta	0.17	0.25	0.31	0.10
Rank	3	2	1	4

According to the previous context, Table 4 summarizes the signal-to-noise (S/N) ratios of an experiment that examined the effects of phosphoric acid concentration, time, power, and nitrogen flow on a process, presumably related to corn cob treatment. Three levels of testing are performed for each factor, resulting in varying S/N ratios. Power is ranked as the most influential factor with a Delta of 0.31, indicating its broadest range of effects on the S/N ratio. Delta 0.25 is the second most important factor, followed by Phosphoric Acid. This table shows that Phosphoric Acid has the smallest range of impact on the process (Delta of 0.17), followed by Nitrogen flow which has the smallest range of impact (Delta of 0.10), and is considered to be the least influential.

Table 5: Response Table for Means

Level	Phosphoric Acid (wt%/v)	Time (min)	Power (KW)	Nitrogen flow (cm3/min)
1	94.29	96.53	95.21	95.36
2	96.18	95.49	93.61	94.67
3	95.32	93.76	96.96	95.75
Delta	1.89	2.78	3.35	1.08
Rank	3	2	1	4

A response table for means is shown in Table 5 and summarizes the average effects of four factors on an experimental response, including Phosphoric Acid concentration, Power, and Nitrogen flow. As an indicator of the impact of each factor on the response, the delta values indicate the range of the mean responses across the levels. A ranking based on deltas shows that Power is the most influential factor, with its Delta of 3.35 (ranked 1); Time is second (ranked 2), Phosphoric Acid is third (ranked 3), and Nitrogen flow is fourth (ranked with a Delta of 1.08). Phosphoric Acid concentration and Time are the factors most likely to influence the outcome of the process, while Nitrogen flow has the least impact.

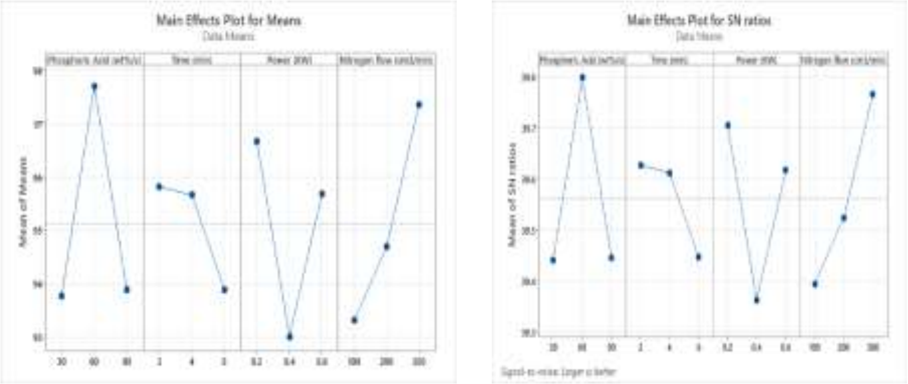


(a) (b)

Fig. 1: Main effect plots of Barley husk for (a) means (b) Signal-to-noise ratio

The graph shown in Fig. 1 (a) illustrates how the response variable is affected by different factors in a Main Effects Plot for Means. Various levels of phosphoric acid concentration, time, power, and nitrogen flow are shown here to show how the mean of a response variable changes. Phosphoric acid concentration and power decrease while time and nitrogen flow tend to increase the mean of the response. Similarly, the graph in Fig. 1 (b) S/N ratios are plotted using Main Effects Plots with the criterion "larger is better" for signal-to-noise ratios. Signal-to-noise ratios are quantified in terms of concentration of phosphoric acid, time, power, and nitrogen flow. As phosphoric acid concentration and power increase, the mean S/N ratio decreases, whereas longer time and higher nitrogen flow result in an increasing trend.

4.2 Taguchi Analysis: corn cob versus Phosphoric Acid (wt%/v), Time (min), Power (KW), Nitrogen flow (cm3/min)



(a) (b)

Fig. 2: Main effect plots of corn cob for (a) means (b) Signal-to-noise ratio

An analysis of Taguchi is presented in the Fig.2. The graph in Fig. 2(a) graph showing the impact of several variables on the treatment of corn cobs is shown in figure 2(a). These variables include the amount of phosphoric acid and the time, power, and flow of nitrogen. Corn cob processing has been shown to be more effective with longer treatment times and increased nitrogen flow than with higher concentrations of phosphoric acid and power. For a corn cob treatment process, this Taguchi Analysis graph in Fig. 2(b) shows signal-to-noise ratios. The process quality is improved when the time and nitrogen flow increase (because "larger is better" for signal-to-noise ratios), while the process quality decreases when the power and concentration of phosphoric acid are increased.

5 Conclusion

The study's application of the Taguchi method has illuminated the pathway to optimizing activated carbon production using corn cob and barley husk as primary raw materials. It revealed that microwave power significantly influences the efficiency and quality of AC, with the concentration of phosphoric acid and the duration of microwave heating also playing essential roles. The research underlines the lesser impact of nitrogen flow rate compared to these factors. By focusing on corn cob and barley husk, agricultural wastes with great potential for AC production, this study contributes to the sustainable and economical production of activated carbon. It addresses not only environmental concerns related to waste management but also supports the increasing demand for effective water treatment solutions.

References

1. Hesas, R. H., Daud, W. M. A. W., Sahu, J. N., & Arami-Niya, A. "The effects of a microwave heating method on the production of activated carbon from agricultural waste: A review." Journal of Analytical and Applied pyrolysis 100 (2013): 1-11.
2. Abioye, Adekunle Moshood, and Farid Nasir Ani. "Recent development in the production of activated carbon electrodes from agricultural waste biomass for supercapacitors: A review." Renewable and sustainable energy reviews 52 (2015): 1282-1293.
3. Heidarinejad, Z., Dehghani, M. H., Heidari, M., Javedan, G., Ali, I., & Sillanpää, M. "Methods for preparation and activation of activated carbon: a review." Environmental Chemistry Letters 18 (2020): 393-415.
4. Sirajo, Lawal, and Muhammad Abbas Ahmad Zaini. "Adsorption of water pollutants using H3PO4-activated lignocellulosic agricultural waste: a mini review." Toxin Reviews 42.1 (2023): 349-361.
5. Alslaibi, T. M., Abustan, I., Ahmad, M. A., & Foul, A. A. "A review: production of activated carbon from agricultural byproducts via conventional and microwave heating." Journal of Chemical Technology & Biotechnology 88.7 (2013): 1183-1190.
6. Taki, Estapraq Ameen, and Sami D. Salman. "A review on Activated Carbon Prepared from Agricultural Waste using Conventional and Microwave Activation." Al-Khwarizmi Engineering Journal 19.3 (2023): 33-43.
7. Ukanwa, K. S., Patchigolla, K., Sakrabani, R., Anthony, E., & Mandavgane, S. "A review of chemicals to produce activated carbon from agricultural waste biomass." Sustainability 11.22 (2019): 6204.
8. Neme, Ibsa, Girma Gonfa, and Chandran Masi. "Activated carbon from biomass precursors using phosphoric acid: A review." Heliyon (2022).
9. Ao, W., Fu, J., Mao, X., Kang, Q., Ran, C., Liu, Y., Zhang, H., Gao, Z., Li, J., Liu, G. and Dai, J.,"Microwave assisted preparation of activated carbon from biomass: A review." Renewable and Sustainable Energy Reviews 92 (2018): 958-979.
10. Foulc, Abu. "A review: production of activated carbon from agricultural byproducts via conventional and microwave heating."
11. Freddi, Alessandro, Mario Salmon, Alessandro Freddi, and Mario Salmon. "Introduction to the Taguchi method." Design principles and methodologies: from conceptualization to first prototyping with examples and case studies (2019): 159-180.

12. Chen, Wei-Hsin, Manuel Carrera Uribe, Eilhann E. Kwon, Kun-Yi Andrew Lin, Young-Kwon Park, Lu Ding, and Lip Huat Saw. "A comprehensive review of thermoelectric generation optimization by statistical approach: Taguchi method, analysis of variance (ANOVA), and response surface methodology (RSM)." Renewable and Sustainable Energy Reviews 169 (2022): 112917.
13. Pradana, Eka Setyo, and Wiwik Sulistiyowati. "Literature Review: Use of the Taguchi Method for Quality Improvement." PROZIMA (Productivity, Optimization and Manufacturing System Engineering) 6, no. 2 (2022): 85-96.
14. Antony, Jiju, Shreeranga Bhat, Anuj Mittal, Raja Jayaraman, E. V. Gijo, and Elizabeth A. Cudney. "Application of Taguchi design of experiments in the food industry: a systematic literature review." Total Quality Management & Business Excellence (2024): 1-26.
15. Chelladurai, Samson Jerold Samuel, K. Murugan, Abhra Pratip Ray, Makarand Upadhyaya, Venugopal Narasimharaj, and S. Gnanasekaran. "Optimization of process parameters using response surface methodology: A review." Materials Today: Proceedings 37 (2021): 1301-1304.
16. Moralı, Uğur, Hakan Demiral, and Sevgi Şensöz. "Optimization of activated carbon production from sunflower seed extracted meal: Taguchi design of experiment approach and analysis of variance." Journal of Cleaner Production 189 (2018): 602-611.
17. Praveena, Sarva Mangala, Umer Rashid, and Suraya Abdul Rashid. "Optimization of nutrients removal from synthetic greywater by low-cost activated carbon: application of Taguchi method and response surface methodology." Toxin Reviews 41, 2 (2022): 506-515.
18. Oemar, Barlin, and Wei-Chin Chang. "Taguchi method for optimizing process parameters in the production of activated carbon from rubber seed shell." The International Journal of Advanced Manufacturing Technology 107 (2020): 4609-4620.
19. Kumar, D. Praveen, D. Ramesh, P. Subramanian, S. Karthikeyan, and A. Surendrakumar. "Activated carbon production from coconut leaflets through chemical activation: Process optimization using Taguchi approach." Bioresource Technology Reports 19 (2022): 101155.
20. Syed-Hassan, Syed Shatir Asghrar, and Mohd Saufi Md Zaini. "Optimization of the preparation of activated carbon from palm kernel shell for methane adsorption using Taguchi orthogonal array design." Korean journal of chemical engineering 33 (2016): 2502-2512.
21. Bhomick, P. C., A. Supong, M. Baruah, C. Pongener, C. Gogoi, and D. Sinha. "Alizarin Red S adsorption onto biomass-based activated carbon: optimization of adsorption process parameters using Taguchi experimental design." International journal of environmental science and technology 17, no. 2 (2020): 1137-1148.
22. Ebrahimi Zarandi, Mohammad Javad, Mahmoud Reza Sohrabi, Morteza Khosravi, Nafiseh Mansouriieh, Mehran Davallo, and Azita Khosravan. "Optimizing Cu (II) removal from aqueous solution by magnetic nanoparticles immobilized on activated carbon using Taguchi method." Water Science and Technology 74, 1 (2016): 38-47.
23. Duran-Jimenez, Gabriela, Virginia Hernandez-Montoya, Miguel A. Montes-Moran, Adrian Bonilla-Petriciolet, and Norma A. Rangel-Vázquez. "Adsorption of dyes with different molecular properties on activated carbons prepared from lignocellulosic wastes by Taguchi method." Microporous and Mesoporous Materials 199 (2014): 99-107.
24. Demiral, İlknur, and Canan Samdan. "Improving Cd+ 2 adsorption capacity of activated carbon by modification method: optimization with taguchi experiment design and analysis of variance." Chemical Papers 76, 4 (2022): 2193-2207.