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Ultrasound-assisted preparation of activated carbon from alkaline impregnated hazelnut shell: An optimization study on removal of Cu^{2+} from aqueous solution

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Abstract

In recent years, the use of new technologies based on non-conventional energies such as ultrasound gains increasing importance. Ultrasound exhibits several beneficial mechanical effects in solid–liquid systems by means of the cavitations phenomenon. In this study, activated carbon adsorbent for removing heavy metals cations such as Cu^{2+} from aqueous solutions has been prepared. For this purpose, hazelnut shells were impregnated with KOH solution under ultrasound irradiation. After filtration, hazelnut shells have been carbonized under inert N_2 atmosphere. The experiments were planned by fractional factorial design and central composite design. Activated carbons were characterized by their copper adsorption capacity. Optimum process conditions were obtained by using a constrained optimization program as follows: particle size 0.83 mm, ultrasound power/volume 19 W/L, impregnation ratio 0.06 g/mL, impregnation time 143 min, activation temperature 838 °C and activation time 19 min, following with maximum adsorption capacity was found as 40 mg Cu^{2+}/g Ac. Activated carbon with the maximum adsorption capacity was further characterized by using scanning electron microscopy and its open pore structure was observed.

Keywords: Statistical modeling; Hazelnut shell; Ultrasound; Activated carbon; Copper adsorption; Optimization

1. Introduction

One of the major methods for the removal of pollutants from aqueous effluent is adsorption by using porous solid adsorbents. Adsorption has demonstrated its efficiency and economic feasibility as a wastewater treatment process compared to the other purification and separation methods, and has gained importance in industrial applications [1,2], such as removal of heavy metals cations from aqueous solution by adsorption onto activated carbon prepared from agricultural wastes [3]. Activated carbon has also found important applications in the field of hydrometallurgy, especially in the recovery of gold and silver from cyanide solutions [4].

Generally, granular activated carbon (GACs) were prepared by activating either physical (dry) method or a chemical (wet) method various carbonaceous raw materials including peat, coal, lignite [5,6] and from cheaper and readily available agricultural by-products such as grape seeds, palm-tree cobs and various nut-shells coconut shell, hazelnut shell, olive-waste cakes and corn cob due to the fact that activated carbon made from conventional raw materials are expensive [2–5,7–11]. In general, the methods for production activated carbon consist of two steps: the first step involves a chemical activation step where raw agricultural materials are impregnated with a solution of dehydrating agent (for example $ZnCl_2$, H_2SO_4) to retard the formation of tars during the carbonization process. Furthermore, in physical activation, they are washed, dried and carbonized in an inert atmosphere to produce the final activated carbon [4,6,8,12].

It is now widely accepted that ultrasound power has great potential for uses, in addition to conventional applications in cleaning and plastic welding, in a wide variety of industrial fields such as electrochemistry, food technology, nanotechnology, chemical synthesis, dissolution and extraction, dispersion of solids, phase separation, water and sewage treatment [13]. Ultrasound produces its mechanical and chemical effects through the formation and collapse of "cavitations" bubbles [14]. A significant amount of research has been published concerning with this "sonochemical effect", and collected in various recent books [15,16]. Ultrasound exhibits also several beneficial mechanical effects in solid–liquid systems by means of

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the cavitations phenomenon; it enhances mass transfer rates, it causes the formation of many micro-cracks on the solid surface, thus, increases the surface area between the reactants, it cleans solid reactant or catalyst particle surfaces [17].

The aim of the present paper is to prepare activated carbon from hazelnut shells to remove Cu^{2+} from aqueous solution. Alkaline impregnation is used as pre-chemical activation and ultrasound irradiation is especially applied in this step to enhance the diffusion of KOH solution into the pores of the cellulosic material. After this step, hazelnut shells are washed, dried and finally carbonized under inert N_2 atmosphere. Activated carbons prepared in this way are characterized by their copper adsorption capacity, scanning electron microscopy and BET surface examination method. Statistical design method is used in the experimentation. The particle size, ultrasound power, impregnation ratio, impregnation time, activation temperature and activation time are chosen as process parameters. The regression model obtained were used in a constrained optimization to find optimum process conditions for maximum adsorption capacity.

2. Experimental

2.1. Experimental design

The orthogonal central composite design was widely applied for fitting a second order model. These designs consist of a 2^n factorial or fractional (coded to the usual ± 1 notation) augmented by $2n$ axial points $(\pm\beta, 0, 0, \ldots, 0), (0, \pm\beta, 0, \ldots, 0), \ldots, (0, 0, \ldots, \pm\beta)$, and m_0 center points (0, 0,0, . . . ,0) [18]. Each variable is investigated at two levels. Meanwhile, as the number of factors, n, increases, the number of runs for a complete replicate of the design increases rapidly. In this case, by assuming high-order interactions negligible, main effects and low-order interactions may be estimated by fractional factorial designs. Individual second order effects cannot be estimated separately by 2^n factorial designs. The first order model is as follows:

$$y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n}\sum_{j>1}^{n} b_{ij} x_i x_j \tag{1}$$

Meanwhile, it is also possible to test the overall curvature generated by pure second order effects by means of the following statistic:

$$\mathrm{LOF}_{\mathrm{curv}} = \frac{m_0 F(\bar{y}_1 - \bar{y}_0)^2}{m_0 + F} \tag{2}$$

where $\bar{y}_0$ is the mean of central replicates and $\bar{y}_1$ is the mean of factorial experiments results. If the variance analysis indicates that overall curvature effect is significant, auxiliary experiments are carried out to develop a second order model. The second order model is defined as follows so as to facilitate calculations:

$$y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii}(x_i^2 - \bar{x}_i^2) + \sum_{i=1}^{n}\sum_{j>1}^{n} b_{ij} x_i x_j \tag{3}$$

where

$$\bar{x}_i^2 = \frac{1}{N}\sum_{i=1}^{N} x_i^2 = \frac{F + 2\beta^2}{N} \tag{4}$$

by defining:

$$b_0' = b_0 - \sum_{i=1}^{n} b_{ii}\bar{x}_i^2 \tag{5}$$

Eq. (3) may be written in usual form as

$$y = b_0' + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n}\sum_{j>1}^{n} b_{ij} x_i x_j \tag{6}$$

Among various second order designs, the orthogonal central composite design is the most popular which requires $2n$ auxiliary runs conducted at two new factor levels, $-\beta$, $+\beta$ [19]. β is calculated by the following relation:

$$\beta = \left(\frac{QF}{4}\right)^{1/4} \tag{7}$$

$$Q = (N^{1/2} - F^{1/2})^2 \tag{8}$$

$$N = 2n + F + m_0 \tag{9}$$

where F is the number of experiments in factorial design, N the total number of experiments and m_0 is the number of central replicates. In the planning and analysis of experimental designs, coded values are usually used instead of absolute values of the variables. The relationship between coded value (X) and absolute value (Z) is as follows:

$$X = \frac{2(Z - Z_0)}{Z_2 - Z_1} \tag{10}$$

where Z_1 is the low level, Z_2 the high level and Z_0 is the medium level of the variable.

2.2. Materials and methods

Hazelnut shells were supplied from the Black Sea Region in Turkey. They were first dried, crushed and sieved to obtain a particle size between 0.78 and 1.85 mm. The experimental set-up consisted of an ultrasonic power generator (Meinhardt ultraschalltechnic, K 80-5, 140 W, 850 kHz), a jacketed glass reactor equipped with a titan probe (E/805/T/solo ultrasonic transducer) which is connected to the bottom of the reactor and fitted with a reflux condenser. A typical impregnation experiment (chemical activation) was carried out as follows: specified particle size and amounts of hazelnut shells and 10% KOH were loaded into the glass reactor and chemical activation process maintained the desired impregnation time. Ultrasound power (continuous mode) is adjusted using the relationship between the intensity setting of the generator, and ultrasound power absorbed by the reaction medium measured by the calorimetric method [20]. Ten percent of KOH solution was used in all the experiments [9,12]. A constant impregnation temperature of 50 °C was applied by means of a constant temperature circulator. At the end of the

impregnation experiments, the sample was immediately filtered, washed with hot distilled water for removal of its alkalinity, the basic and water-soluble components and dried. The impregnated sample was carbonized in a furnace (Carbolite, CWF 1300) under N_2 atmosphere (1 kg/cm^2) at desired carbonization temperature and time for final activated carbon. The surface area of the activated carbon samples are investigated by using JEOL (JSM) 6400 scanning electron microscopy and the BET surface area method by a Quantachrome QS-17 model apparatus.

2.3. Determination of the copper adsorption capacity and adsorption isotherm

Batch mode adsorption experiments were carried out in a shaker Thermolyne Rosi 1000^{TM} (Reciprocating/Orbital Shaking Incubator) Model. Hundred millgram per liter solutions of Cu^{2+} were prepared by dissolving the solid $CuSO_4{\cdot}5H_2O$ in distilled water. Samples of 200 mg of activated carbon was added to 100 mL solution of Cu^{2+} in 250 mL erlenmayer flasks and shaked at 18 °C, 170 rpm for a contact time of 6 h which was found sufficient to obtain a nearly constant adsorption capacity in the light of pre-experiments. The pH of the Cu^{2+} solution was not adjusted and activated carbon added to the Cu^{2+} solution was not changed the pH of the Cu^{2+} solution. At the end of the experiments, the solutions of Cu^{2+} were separated from the samples activated carbons by filtering and filtrates were analyzed by using a Atomic Absorption/Flame Emission Spectrophotometer Shimadzu Model AA-670.

3. Results and discussion

3.1. Response analysis and interpretation

The particle size (X_1), ultrasound power (X_2), impregnation ratio (X_3), impregnation time (X_4), activation temperature (X_5) and activation time (X_6) were chosen as the independent parameters by taking into account the pre-experiments for modeling of Cu^{2+} adsorption from aqueous solutions. The parameter levels and coded values were given in Table 1.

The 2^{6-2} orthogonal fractional factorial and central composite designs were applied to estimate main effects and second order effects as well as interaction effects. Furthermore, three central replicates were also employed to calculate pure experimental error. As usual, the experiments were carried out in a random order to minimize the effect of systematic error. The experimental design matrix and the corresponding experimental parameters and response value were shown in Table 2. In order to model experimental results, Matlab statistical toolbox was used. The full second order model relating process response to process parameters obtained by regression analysis is as follows:

$$\begin{aligned} Y_{Cu^{2+}} = {} & 20.5558 - 0.5950X_1 - 0.1441X_2 - 0.1553X_3 \\ & + 0.1902X_4 + 1.1670X_5 - 0.6848X_6 + 0.2882X_1^2 \\ & + 1.0302X_2^2 - 0.2687X_3^2 - 0.0950X_4^2 - 0.2162X_5^2 \\ & + 0.5619X_6^2 + 0.1969X_1X_2 + 0.3053X_1X_3 \\ & + 0.6945X_1X_4 - 2.1615X_1X_5 - 0.5734X_1X_6 \\ & - 2.1615X_2X_3 - 1.6740X_2X_4 + 0.3053X_2X_5 \\ & - 0.1781X_2X_6 - 0.1781X_3X_4 + 0.1969X_3X_5 \\ & - 1.6740X_3X_6 - 0.5734X_4X_5 - 2.1615X_4X_6 \\ & + 0.6945X_5X_6 \end{aligned} \quad (11)$$

This model is 27 factors, estimates the experimental data very well and the correlation coefficient (r^2) of model obtained in this case is 0.9999. However, a simpler model with fewer factors and with a high r^2 value may also be established by means of variance analysis. For this purpose, the model established with effective parameters obtained by variance analysis conducted at 90% confidence level was given as follows. The correlation coefficient (r^2) of model obtained in this case is 0.9164:

$$\begin{aligned} Y_{Cu^{2+}} = {} & 20.7499 - 0.5950X_1 + 1.1670X_5 - 0.6848X_6 \\ & + 1.0302X_2^2 + 0.6945X_1X_4 - 2.1615X_1X_5 \\ & - 2.1615X_2X_3 - 1.6740X_2X_4 - 1.6740X_3X_6 \\ & - 2.1615X_4X_6 + 0.6945X_5X_6 \end{aligned} \quad (12)$$

In this model as a simpler model with fewer factors (l1 factors) for a well-established, systematic errors are absent, and normalized residuals result from experimental errors which exhibit a normal distribution according to a widely accepted statistical convention. Fig. 1 shows the graphical representation of 'size effect' of each parameters upon the activated carbon prepared from ultrasound-assisted KOH-impregnated hazelnut shell as adsorbents to remove Cu^{2+} from aqueous solutions. From Fig. 1, it can be seen that activation temperature (X_5) has a positive effect, while particle size (X_1) and activation time (X_6) have a negative effect on response, in the way of amount of the adsorbed mg Cu^{2+}/g Ac from aqueous solutions. The second order term and interaction terms in Eq. (12) affect to process at various ratios.

Table 1
Parameter levels (coded values) used in the experimental design

Parameter	$+\beta$	+1	0	−1	$-\beta$
Particle size, X_1 (mm)	1.85	1.55	1.29	0.93	0.78
Ultrasound power/volume, X_2 (W/L)	190	76	19	6	2
Impregnation ratio, X_3 (g/mL)	0.06	0.05	0.038	0.025	0.015
Impregnation time, X_4 (min)	143	120	90	60	37
Activation temperature, X_5 (°C)	839	800	750	700	661
Activation time, X_6 (min)	72	60	45	30	18

Table 2
Experimental design matrix and response value

Exp. no.	Particle size	Ultrasound power	Impregnation ratio	Impregnation time	Activation temperature	Activation time	Adsorbed (mg Cu^{2+}/g Ac)
3	−1	1	−1	−1	1	1	26
4	1	1	−1	−1	−1	1	24
15	−1	1	1	1	−1	1	11
10	1	−1	−1	1	1	1	21
8	1	1	1	−1	1	−1	20
6	1	−1	1	−1	−1	1	16
11	−1	1	−1	1	1	−1	27
5	−1	−1	1	−1	1	1	26
16	1	1	1	1	1	1	22
9	−1	−1	−1	1	−1	1	24
1	−1	−1	−1	−1	−1	−1	15
13	−1	−1	1	1	1	−1	25
14	1	−1	1	1	−1	−1	28
2	1	−1	−1	−1	1	−1	24
12	1	1	−1	1	−1	−1	22
7	−1	1	1	−1	−1	−1	21
31	−1.77	0	0	0	0	0	26
23	1.77	0	0	0	0	0	17
22	0	−1.77	0	0	0	0	24
21	0	1.77	0	0	0	0	24
28	0	0	−1.77	0	0	0	17
25	0	0	1.77	0	0	0	23
29	0	0	0	−1.77	0	0	22
27	0	0	0	1.77	0	0	18
24	0	0	0	0	−1.77	0	21
26	0	0	0	0	1.77	0	19
30	0	0	0	0	0	−1.77	23
20	0	0	0	0	0	1.77	21
1^0	0	0	0	0	0	0	21
2^0	0	0	0	0	0	0	20
3^0	0	0	0	0	0	0	22
Raw, Hazelnut, Shell							2

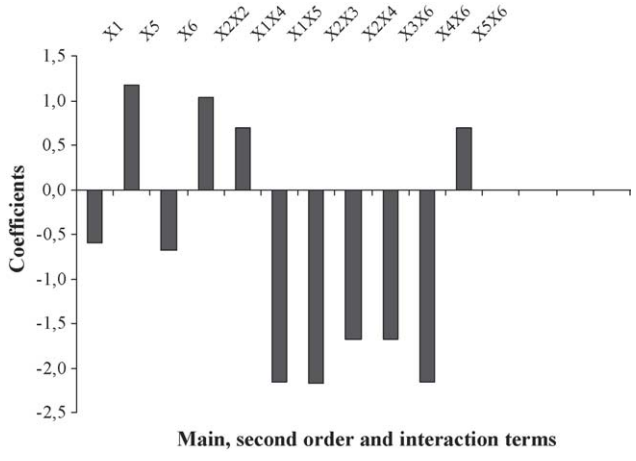


Fig. 1. Significant main, second order and interaction terms on the activated carbon prepared from ultrasound-assisted KOH-impregnated hazelnut shell as adsorbents to remove Cu^{2+} from aqueous solutions.

3.2. Optimization results

The main objective of the optimization is to determine the optimum process conditions from the models obtained with theoretically or experimentally. For this purpose, firstly, the objective of the optimization is decided, which may be either economic or technical. Secondly and last, the main objective of this research is to determine the experimental conditions required to prepare activated carbon from hazelnut shell, suitable for their efficient employment as adsorbents to remove Cu^{2+} from aqueous solutions. Eventually, by using above-mentioned methodology for experimental design, the ranges of the parameters required to obtain this optimum activated carbon were determined. In this optimization study, amount of the adsorbed mg Cu^{2+}/g Ac from aqueous solutions were chosen as the objective function. Furthermore, optimum conditions are often calculated in the presence of some constraints which ensure them to be more realistic. If the model used in the optimization study is an empirical one, high and low levels of the process parameters in the experimental design are considered, inevitably, as explicit constraints, in order to avoid extrapolation.

Thus, the optimization problem is defined as

$$\text{maximize } Y_{Cu^{2+}} \tag{13}$$

Constraints on the parameters X:

$$-\beta_i < X_i < +\beta_i, \quad i = 1, \ldots, 6 \tag{14}$$

$-\beta$ and $+\beta$ values are given in Table 1. The optimization problem given in Eq. (13) is solved using constrained optimization

Table 3
The optimum process conditions to prepare activated carbon from hazelnut shell

Particle size (mm)	0.83
Ultrasound power/volume (W/L)	19
Impregnation ratio (g/mL)	0.06
Impregnation time (min)	143
Activation temperature (°C)	838
Activation time (min)	19
Adsorbed (mg Cu^{2+}/g Ac)	40

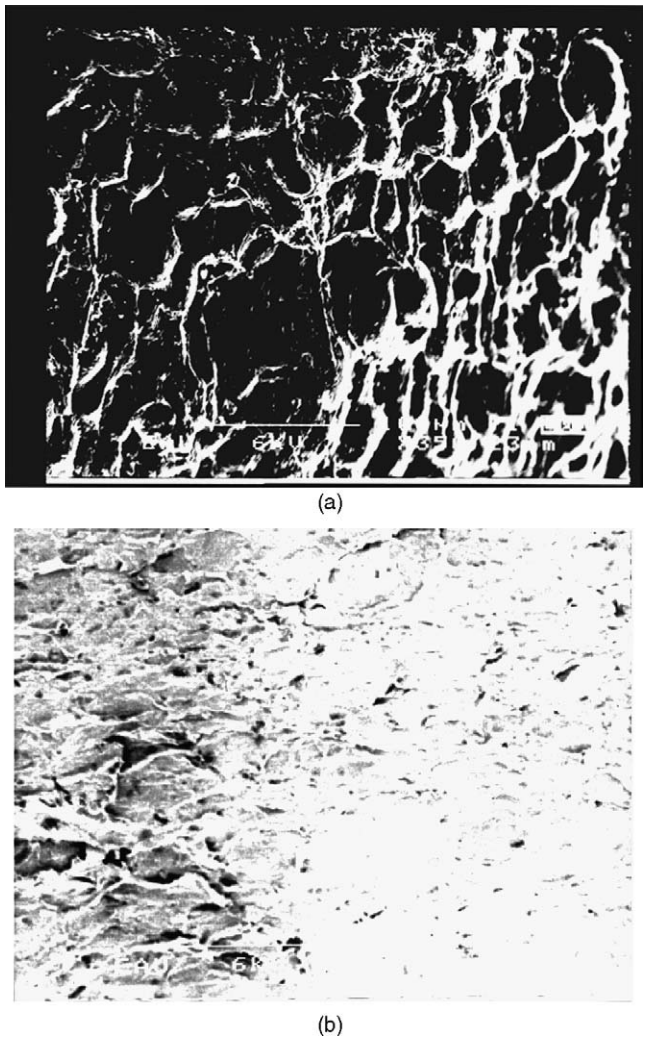
program supplied in the Matlab optimization toolbox. These results show that ultrasound power is effectively used at its lower bound, high impregnation ratio and time are sufficient for optimum activated carbon from hazelnut shell for use as adsorbents to remove Cu^{2+} from aqueous solution, whereas particle size, carbonization temperature and time have more strong impacts on the optimum yield. The optimum process conditions are given in Table 3 by taking into account the model, which is established with effective parameters obtained by variance analysis conducted at 90% confidence level. Maximum adsorption capacity of activated carbon from alkaline impregnated hazelnut shell under ultrasound is 40 mg Cu^{2+}/g Ac, whereas maximum adsorption capacity of activated carbon from alkaline impregnated hazelnut shell under the same process conditions given in Table 3, but without ultrasound application has been found as 22 mg Cu^{2+}/g Ac. According to these results, the adsorption capacity of the activated carbon from alkaline impregnated hazelnut shell under ultrasound is approximately two times greater than that of the adsorption capacity of activated carbon from alkaline impregnated hazelnut shell without ultrasound. In conclusion, the application of ultrasound irradiation in the impregnation step is found to be beneficial to prepare with high adsorption capacity activated carbon for use as adsorbent to remove Cu^{2+} from aqueous solutions.

3.3. Surface characterization of the optimally prepared activated carbon

The shapes of adsorption isotherms can provide qualitative information on the adsorption process and the extent of the surface area available to the adsorbate. Adsorption isotherm for copper from aqueous solutions was also determined by contacting 200 mg of the activated carbon sample with 100 mL solution of Cu^{2+} of varying initial concentrations. The Langmuir and Freundlich isotherms constants were calculated for adsorption of Cu^{2+} from in different initial concentrations of aqueous solutions. The Langmuir and Freundlich isotherms constants are listed in Table 4. Error estimation of the Langmuir and Freundlich equations show that Freundlich equations is more accurate to describe the Cu^{2+} adsorption isotherm than Langmuir equation.

Table 4
Langmuir and Freundlich constants for the Cu^{2+} adsorption

Langmuir constant		Freundlich constant		Equilibrium range (ppm)
Q_{max} (mg/g)	b (L/mg)	K_F (L/g)	n	
39.54	0.125	12.544	4.42	10–198


(a)

(b)

Fig. 2. (a) The surface of the activated carbon with maximum adsorption capacity; (b) the surface of activated carbon adsorbed Cu^{2+} from aqueous solution.

The SEM photographs of the surface appearances of the activated carbon are shown in Fig. 2a and b. Fig. 2a shows the surface of activated carbon prepared by ultrasound-assisted KOH impregnated (chemical activation) and carbonized (physical activation) at the experiment conditions in a furnace under N_2 atmosphere at desired carbonization temperature and time. It can be seen from the photograph that the external surface of this carbon is full of cavities because of mechanical effects of ultrasound. It seems that the cavities on the surface resulted from the evaporation of KOH during carbonization, leaving the space previously occupied by KOH. Fig. 2b shows the surface of activated carbon which adsorbed Cu^{2+} from aqueous solution. It seems that the cavities on the surface are occupied full of copper. BET surface areas of the raw and activated carbon from hazelnut shell were calculated to be 0.188 and 10.1 m^2/g, respectively. Considering BET surface areas, the surface area of the activated carbon is 50 times greater than raw hazelnut shell surface area. In conclusion, it is considered that the surface area and adsorption capacity can be increased for activated carbon

prepared from ultrasound-assisted KOH-impregnated hazelnut shell for use in wastewater treatment.

4. Conclusions

The combined chemical and physical activation process was attempted to prepare activated carbon from ultrasound-assisted KOH-impregnated hazelnut shell for use in water treatments. For this purpose, the experimental parameters, the particle size, ultrasound power, impregnation ratio, impregnation time, activation temperature and activation time have been explored by statistically designed experiments. The 2^{6-2} orthogonal fractional factorial design and central composite design were applied to establish second order model relating amount of the adsorbed mg Cu^{2+}/g Ac from aqueous solutions to experimental parameters and the optimal experimental conditions required to produce suitable activated carbon for the use of adsorbents to remove Cu^{2+} from aqueous solutions were obtained. In conclusion, the application of ultrasound irradiation in the impregnation step is found quite beneficial to increase adsorption capacity of activated carbon for use as adsorbent. However, detailed optimization researches along with cost analysis are needed to assess the profitability of the method. It is expected that the optimization results presented in this paper may provide background information for a detailed process improvement research.

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