

Rhodium Removal from Wastewater

Firas Saeed Abbas

Environmental Engineering Department, Faculty of Engineering, Al-Mustansiriyah University, Baghdad, Iraq

ABSTRACT

Rhodium removal from aqueous solutions using different design parameters by adsorption process using banana peel. The design parameters studied to adsorb rhodium using BP as an adsorbent material were initial concentration of rhodium, adsorbent material packing height which was BP, pH of rhodium feed inlet, treatment time, feed flow rate and feed temperature, these parameters were varied from (1-100) mg/l, (10-100) cm, (1-8), (1-60) min, (5-100) ml/min and (20-45°C) respectively. Results show that the higher removal efficiency was 96.15 % for rhodium from aquatic solution and this efficiency was decreased with increasing of initial concentration and flow rate while the removal efficiency increased with increasing pH, sorption media bed height and feeding temperature. A mathematical model was proposed for an adsorption packed bed column with porous adsorbent. The mathematical model takes account of both external and internal mass-transfer resistances as well as of nonideal plug flow along the column. Statistical model is achieved to find an expression combined all operating parameters with the removal efficiency for rhodium used in this paper in a general equation. The samples of BP remaining after using it in the removal of rhodium above from aqueous solutions aforementioned were predestined to investigate the utilization of it in useful method. One of these methods was using it as a promoter in preparing type Y-zeolite catalyst, compared these type prepared above with normally type Y-zeolite catalyst and tested in the process of n-heptane catalytic cracking under different operating conditions. The results show that the promoted type Y-zeolite catalyst by rhodium gives higher conversion and better selectivity of n-heptane catalytic cracking than normally type Y-zeolite catalyst.

Keywords: Banana Peel, Rhodium, Aqueous Solutions, Adsorption, Residue, Wastewater

I. INTRODUCTION

Rhodium, compose together with ruthenium, palladium, osmium, iridium and platinum an element group named platinum group metals (PGM) ⁽¹⁾. Rhodium founds as rare sediment of the uncomposite metal. Commercially, the metal is produced from the by-product refining of nickel and copper ores which can contain up to 0.1% rhodium ⁽²⁾. Rhodium metal is silvery white and brilliant, has a melting point higher than platinum and lower density than it. Its reflectance is high and it's hard and sturdy. When calefactory, rhodium divert to oxide state when red and at higher temperatures divert again to the element. Rhodium not reacts with air and water above 600°C. Rhodium explosion is possible if it is in granular and powder forms (*Ngwenya, 2005*). The main use of rhodium metal is to produce alloys with platinum and iridium, because of its high-temperature, strength and oxidation resistance properties.

These alloys are used in furnace windings, pen nibs, phonograph needles, high-temperature thermocouple and resistance wires, electrodes for aircraft spark plugs, bearings and electrical contacts. Catalytic converters for cars take (85%) for rhodium metal using. The metal itself is used to plate jewellery and the reflectors of searchlights, due to its brilliance and resistance to tarnish. It is also a highly useful catalyst in a number of industrial processes, such as the petroleum process industries. The uses of rhodium are distributed among the chemical (38%), electrical (25%), jewellery (17%) and glass (12%) industries (*Ngwenya, 2005 and ACGIH, 1981*). Rhodium compounds stain the skin very strongly and caused nasal irritation the concentration of rhodium salt was in the order of 0.002 mg Rh/m³ for the workers in plants. All rhodium compounds must be considered as very toxic and as carcinogenic factors (*Goering, 1992*). The aim of this investigation is studying the ability of banana peel (low

coast adsorbent material) for removing rhodium from simulated synthetic aqueous solution, and utilization from the banana peel residue in eco-friendly method.

II. EXPERIMENTAL WORK

Fixed bed column of continuous mode experiments were conducted in order to examine rhodium removal by treated SSAS of above rhodium at desired concentration with the various bed heights of the BP (adsorbent media) using different flow rates of SSAS of rhodium at various pH. The pH value was adjusted using 0.1 N NaOH and 0.1 N HCl solutions. The sorption unit consists of two glass container for SSAS of rhodium one for inlet feed and another for outlet each of (1 liter) capacity. Glass column has 2.54 cm ID and 150 cm height. The sorption column packed with adsorbent media to a height of (10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 cm) supported from the top and the bottom by glass hollow cylinder layer, each cylinder have (0.5 cm ID, 0.1 cm thickness and 1 cm long). Before starting the runs, the packed bed sorption column was rinsed by double distilled water down flow through the column. The adsorbent media is packed in the column to the desired depth, and fed to it as slurry by mixing BP with distilled water in order to avoid the formation of air bubbles inside the media. After the packed bed sorption column was accommodation and putting the required amount of adsorbent media, the adsorption process started by allowing the rhodium SSAS of required concentration and pH down flow through the sorption column from inlet container by gravity at a precise flow rate in experiment which is adjusted by rotameter. To determination the best operational conditions, the experiments were carried out at a temperature between (20-45°C), various pH values which are (1-8) and initial feed concentrations of SSAS of different rhodium which are between (1-100) mg/l each one alone and at different flow rates which are between (5-100) ml/min for SSAS of rhodium initial feed concentration. Outlet samples after treatment in each experiment were collected every 10 minutes from the bottom of packed column and the unabsorbed concentration of rhodium in SSAS was analyzed by spectrophotometer.

In order to check the reusability of sorbent media, the treated BP with different rhodium concentrations was firstly dried and tested again in sorption unit at the conditions of experiment gives the best percent removal

of rhodium from SSAS aforementioned. The capacity of the sorbent was found to be decreased until be constant at destined percent removal after different times repeated use. The destined percent removal and number of repeated uses were dependent on rhodium concentrations; thus *eleven times* of sorbent use were seen to be feasible.

III. MATHEMATICAL MODEL

Fixed bed dynamics are describing by a set of convection-diffusion equations, coupled with source terms due to adsorption and diffusion inside adsorbent particles. Inside the particle, molecules of adsorbate diffuse into the inner portions of particle via surface diffusion, pore diffusion, or both. The solution of these equations will give rise to the prediction of the needed concentration distribution. This investigation focuses on understanding the mechanism of both surface diffusion and pore diffusion. To formulate a generalized model corresponding to the dispersion flow, surface diffusion and pore diffusion mechanism, following assumptions are made:

1. The system operates under isothermal conditions.
2. The equilibrium of adsorption is described by Langmuir isotherm.
3. Intraparticle mass transport is due to Fickian diffusion, and it is characterized by the pore diffusion coefficient, D_p and the surface diffusion, D_s .
4. Mass transfer across the boundary layer surrounding the solid particles is characterized by the external-film mass transfer coefficient, k_f .
5. Film transfer resistance for mass transport from the mobile to the stationary phase.
6. Local adsorption equilibrium between the adsorbate adsorbed onto the adsorbent surface and the adsorbate in the intraparticle stagnation fluid.
7. Both surface and pore diffusion are included in the mass transport mechanism.
8. Axial dispersion.

Simulation Technique

Equations used in simulation technique represent a set of simultaneous, nonlinear, partial differential equations (PDEs) that can be solved numerically. The discretization was applied to space coordinates (Z and r) to convert the PODs to a set of ordinary differential equations (ODEs). The resulting ODEs can be solved

using an existing ODE solver provided by MATLAB (Cesar, 1999).

IV. RESULTS AND DISCUSSION

The ability of BP to remove rhodium from SSAS in fixed bed column of continuous mode at various parameters which are pH's of SSAS of rhodium (pH), height bed of adsorbent media BP (h), flow rates of SSAS (F), SSAS temperature (T_{feed}) and time of treatment (t) was investigated. The experiments were achieved by varying all above parameters for different initial concentrations (C_o) of SSAS of rhodium.

4.1: Effect of Initial Concentration

The results showed that using adsorbent material, the percent removal of rhodium was decreased when the initial concentration (C_o) of SSAS of rhodium was increased at constant other variables as shown in Figure 1. This can be explained by the fact that the initial concentration of rhodium had a restricted effect on rhodium removal capacity; simultaneously the adsorbent media had a limited number of active sites, which would have become saturated at a certain concentration. This was lead to the increase in the number of rhodium molecules competing for the available functions groups on the surface of adsorbent material.

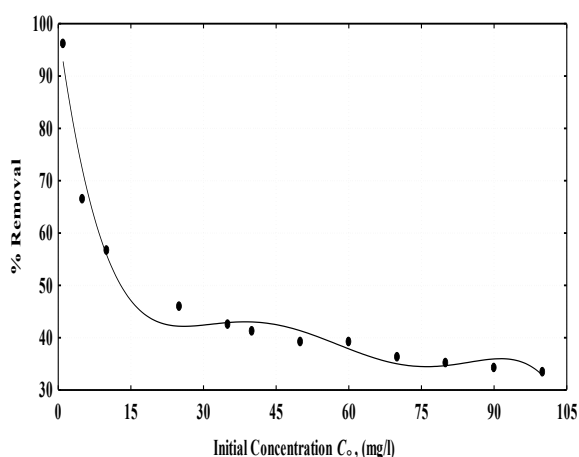


Figure 1: Effect of initial concentration (C_o) on the percent removal of rhodium @ $T_f=45^\circ\text{C}$, $l = 1 \text{ m}$, $pH=8$, $t=60 \text{ min.}$ and $F=5 \text{ ml/min.}$

Since the solution of lower concentration has a small amount of rhodium than the solution of higher concentration of it, so the percent removal was decreased with increasing initial concentration of rhodium. For adsorbent media, higher percent removal

was 96.51 % for rhodium at initial rhodium concentration of 1 mg/l, so adsorbent material was found to be efficient to rhodium removal from SSAS and wastewater.

4.2: Effect of pH

The results showed that using adsorbent material, the percent removal of rhodium was increased when the pH of SSAS of rhodium was decreased at constant other variables as shown in Figure 2. It is well recognized that the pH of the SSAS is an important parameter in affecting adsorption of rhodium⁽⁹⁾. High adsorption of rhodium ions at low pH can be explained in both terms; the species of rhodium and the adsorbent surface. For this case, at low pH, i.e. acidic conditions, the surface of the adsorbent (BP) becomes highly protonated and favours adsorb of above group of rhodium in the anionic form.

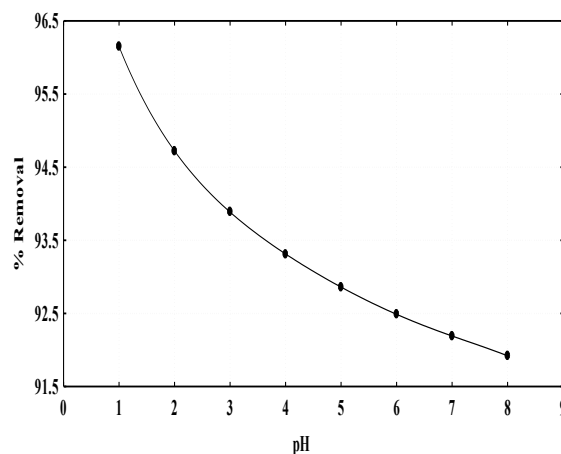


Figure 2: Effect of pH on the percent removal of rhodium @ $C_o = 1 \text{ mg/l}$, $T_f=45^\circ\text{C}$, $l = 1 \text{ m}$, $t=60 \text{ min.}$ and $F=5 \text{ ml/min.}$

With increasing the pH of aqueous solution, the degree of protonation of the surface reduces gradually and hence adsorption is decreased⁽¹⁰⁾. Furthermore, as pH increases there is competition between hydroxide ion (OH^-) and metal ions, the former being the dominant species at higher pH values. The net positive surface potential of sorbent media decreases, resulting in a reduction the electrostatic attraction between the (sorbent) rhodium ion species and the (sorbate) adsorbent material surface (BP), with a consequent reduced sorption capacity which ultimately leads to decrease in percentage adsorption of rhodium ion (Abbas and Abbas, a, 2013; Abbas and, Abbas, b, 2013).

4.3: Effect of Adsorbent Media Bed Height

The results elucidated that when the adsorbent media bed height was increased, the percent removal of rhodium was increased too at constant other variables as shown in **Figure 3**. The increased of bed height meaning increased in the amount of adsorbent media BP, thus increasing the surface area of adsorbent material, hence increased the number of active sites in the adsorbent material surface i.e. increased the availability of binding sites for adsorption and consequently increase rhodium removal capacity on BP.

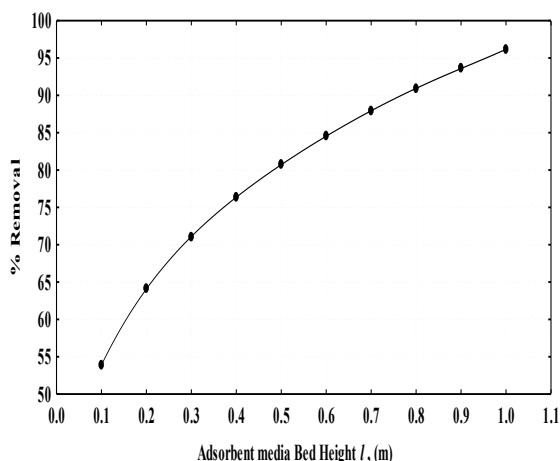


Figure 3: Effect of adsorbent media bed height (l) on the percent removal of rhodium @ $C_o = 1$ mg/l, pH=8, $T_f = 45^\circ\text{C}$, $t = 60$ min. and $F = 5$ ml/min.

This lead to increase the ability of adsorbent media to adsorb greater amount of rhodium from SSAS at different initial concentrations and ultimately the percent removal of rhodium increased.

4.4: Effect of Flow Rate

The results illustrated that when the flow rate of SSAS of rhodium was increased, the percent removal of rhodium was decreased at constant other variables as shown in **Figure 4**. This may be due to the fact that when the flow of SSAS of rhodium increasing, the velocity of SSAS in the column packed with the adsorbent media BP was increasing too, so the solution spend shorter time than that spend in the column while at low flow rate, and the SSAS of rhodium resides in the column for a longer time, therefore undergoes more treatment with the adsorbent media, thus the adsorbent media uptake low amount of rhodium from SSAS of rhodium for high flow rate, therefore the percent

removal of rhodium was decreased when the flow rate was increased.

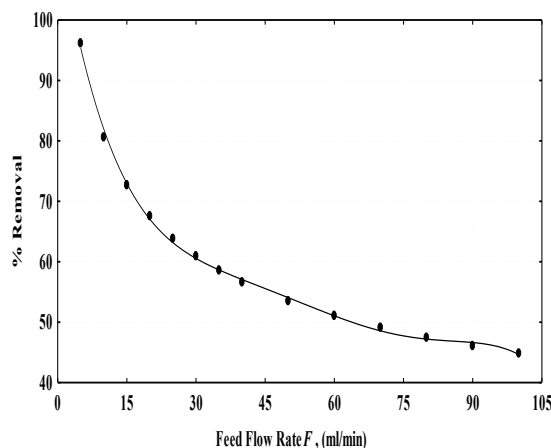


Figure 4: Effect of SSAS flow rate (F) on the percent removal of rhodium @ $C_o = 1$ mg/l, pH=8, $T_f = 45^\circ\text{C}$, $l = 1$ m and $t = 60$ min.

4.5: Effect of Feed Temperature

The results demonstrated that when the temperature of feed which was SSAS of rhodium was increased, the percent removal of rhodium was increased too at constant other variables as shown in **Figure 5**. The effect of temperature is fairly common and increasing the mobility of the acidic ion. Furthermore, increasing temperatures may produce a swelling effect within the internal structure of the adsorbent media enabling rhodium ions (Rh^{+3}) to penetrate further. It was indicated that rhodium adsorption capacity increased with increasing feed temperature from 20 to 45°C .

This effect may be due to the fact that at higher temperature an increase in active sites occurs due to bond rupture.

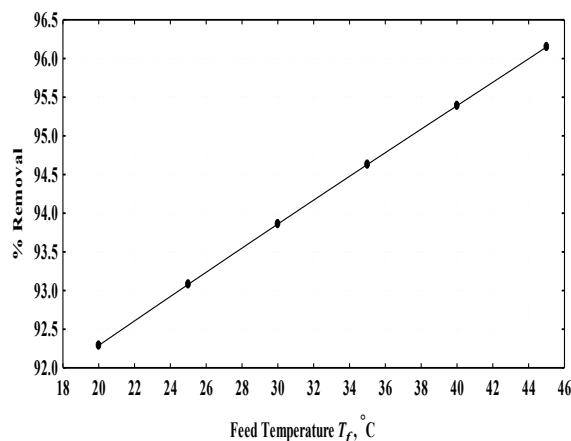


Figure 5: Effect of feed temperature (T_f) on the percent removal of rhodium @ $C_o = 1$ mg/l, pH=8, $l = 1$ m, $t = 60$ min. and $F = 5$ ml/min.

4.6: Effect of Treatment Time

The results demonstrated that when the treatment time of SSAS of rhodium increased the percent removal of rhodium increased at constant other variables as shown in **Figure 6**. This may be due to the fact that when the treatment time of rhodium SSAS increasing and the velocity of SSAS in the column packed with the adsorbent material was remaining constant, the solution spend longer time than that spend it when the time of treatment decreased, so the adsorbent material uptake more amount of rhodium from SSAS, therefore the percent removal of rhodium from SSAS was increased.

V. STATISTICAL MODEL

A statistical model was carried out to the experimental results obtained from this study. Regression analysis and π Theorem was adopted to maintain a relation between the percent removal of rhodium and the feed temperature, flow rate, pressure, pH of feed solution, initial concentration of rhodium, adsorbent media of BP bed height, treatment time, column diameter and other parameters. These relations are shown in **equation 1** below, which has a correlation coefficient (R^2) 0.9991.

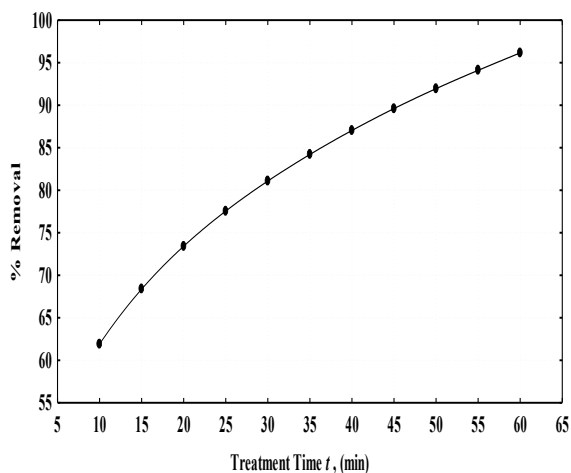


Figure 6: Effect of treatment time (t) on the percent removal of rhodium @ $C_o = 1$ mg/l, $T_f = 45^\circ\text{C}$, $\text{pH} = 8$, $l = 1$ m, and $F = 5$ ml/min.

VI. INVESTMENT OF BANANA PEEL

Huge amount of BP was lingered after using it in removal of rhodium from SSAS as explained above. Utilization from BP uses can be achieved as a promoter of zeolite catalyst follows:

6.1: Employment of used banana peel to prepare promoted zeolite type Y catalyst

BP waste adsorbed rhodium from SSAS at different operating conditions were segregated and classified according to its contain of rhodium and utilization from these remaining samples as a promoter for catalyst used in n-heptane cracking: zeolite type Y. The samples selected give different ratios of rhodium to BP. The ratios were between (0.1 to 0.5 wt %) for rhodium adsorb. Rice husk (which was a raw material for zeolite type Y catalyst synthesis) firstly treated with 10% phosphoric acid (H_3PO_4) for 24 hours.

Then they were well washed with double distilled water, filtered, dried in air, and calcined at 750°C for 6 hours. 12 g of calcined rice husk were then subjected for dissolution in sodium hydroxide NaOH (4 M) followed by refluxing at 90°C for 12 hours. After that concentrated hydrochloric acid (HCl (37%)) was added to the aforementioned base dissolved rice husk for complete precipitation. Rice husks were filtered, washed with excess distilled water to be freeing from chloride ions and finally dried in an oven at 120°C for 6 hours. Zeolite type Y was synthesized using prepared rice husk above as a silica source in the following method. A 500 ml Teflon beaker containing a magnetic stirrer was washed with deionized water.

Sodium hydroxide of 1.6616g was added slowly to deionized water and stir until clear and homogenous solution appeared for about 5 minutes. The aqueous solution of sodium hydroxide was ready for the preparation of seed gel. The gel was prepared according to the following molar chemical composition: $10.67 \text{ Na}_2\text{O} : \text{Al}_2\text{O}_3 : 10 \text{ SiO}_2 : 180 \text{ H}_2\text{O}$. Two millilitres aqueous solution of sodium hydroxide was added to 0.7515g sodium aluminate oxide until a homogenous mixture was formed; 1.5361g of prepared rice husk above was added separately to 5.5 ml sodium hydroxide aqueous until mixed homogeneously. Both of the preparations were heated under vigorous stirring to obtain a homogenous mixture.

The sample was aged for 24 hours at room temperature in the Teflon bottle. The aluminate and silicate solutions were mixed together in the polypropylene beaker, subsequently stirred for 2 hours with the purpose of making it completely homogenized. This combined

solution was used as the feed stock gel. The synthesized zeolite type Y which was in sodium (Na⁺) powder form. In order to make a promoted catalyst (Rh/HY-zeolite) ready for test in any process, hydrogen zeolite (HY-zeolite) form must be prepared.

$$\%R = 1.761 \times 10^{-7} \left(\frac{\rho_{sol}}{C_o}\right)^{0.2295} \cdot \left(\frac{l}{d}\right)^{0.2516} \cdot \left(\frac{T_f \cdot C_{P_{sol}} \cdot t}{\vartheta}\right)^{0.2458} \cdot \left(\frac{K_{sol} \cdot T_f}{\gamma \cdot u}\right)^{0.2547} \cdot \left(\frac{1}{pH}\right)^{0.0217} \dots (1)$$

where: %R Percent Removal of Rhodium from SSA

ρ_{sol} Density of SSAS, (kg/m³)

C_o Initial Concentration of Rhodium, (kg/m³)

l Adsorbent Material Bed Height, (m)

d Internal Diameter of Sorption Column, (m)

T_f Feed Temperature, (K)

$C_{P_{sol}}$ Heat Capacity of SSAS, (kJ/kg. K)

t Treatment Time, (s)

ϑ kinematic viscosity of SSAS, (m²/s)

K_{sol} Thermal Conductivity of SSAS, (W/m. K)

γ Surface Tension, (N/m)

u Velocity of SSAS, (m/s)

The HY-zeolite was prepared by exchanging Na⁺ ions in the sodium form zeolite type Y with ammonium chloride solution NH₄Cl. In order to obtain ideal degree of ion exchange the technique of multi-steps (three times repeating) was used. Thus, the first step, 2N of ammonium chloride solution (26.75 g of NH₄Cl in 250 ml of distilled water) contacted with 90 g of prepared NaY-zeolite with stirring for 2 hours. In the second step, the procedure in the first step was repeated under the same conditions but on about 60 g of zeolite, which was taken from the total zeolite amount produced in the first step. Finally, in the third step, the procedure under the same conditions was repeated again but on about 30 g of zeolite, which was taken from the total zeolite amount produced in the second step. The exchanged ammonia zeolite were filtered off, washed with deionized water to be free of chloride ions dried overnight at 120°C and then calcined initially at 150°C for two hours. The temperature was increased 75°C per hour until it reached 550°C and it was held constant for 5 hours at this temperature. During calcination, ammonia and water were liberated and HY-zeolite was formed (*Abbas and Abbas, c, 2013*). A 25 g of prepared HY-zeolite powder was used for the preparation of Rh/HY-zeolite by impregnation method. 1.3688 g of HCl was mixed with a solution of waste BP using in adsorbing rhodium and water. The resultant solution is added drop by drop to 25 g of prepared HY-zeolite with continuous agitation. Then, the sample mixed by using a magnetic

stirrer to have a homogeneous distribution of rhodium and, the slurry was dried at 110°C overnight and calcined at 400°C for 5 hours. The calcined catalyst was reduced with hydrogen at 380°C for 4 hours with hydrogen. The prepared catalyst powder was mixed with 15 % montmorillonite clay as a binder and water to form a paste. A spherical shape (nearly 0.5 cm) was formed, dried overnight at 110°C (*Abbas, 2004*) and promoted zeolite which is Rh/HY-zeolite catalyst was synthesis.

6.1.1: Catalytic Activity of Synthesized Catalysts

The promoted and normally type Y zeolite catalyst activity was studied by applying n-heptane catalytic cracking reaction. The n-heptane catalytic cracking reaction was carried out in catalytic cracking unit which is operated under atmospheric pressure and the experiments of catalytic cracking were performed in an experimental fluidized bed unit. The unit consists of n-heptane storage tank, gas flow meter, dosing pump, evaporator, condenser/separator, cooler with appropriate control, and power supply box. A suitable amount of Rh/HY-zeolite catalyst prepared above was charged in the reactor from the top of it. At preheating section, the feed was preheated at 400°C before entering to catalytic bed. The temperature controlled was setting at desired reaction temperature between (400-550°C) in the reaction zone. The first condenser was supplied with water from chiller and second charged with ice. A suitable quantity of water was charged to the preheater to remove air from the reactor. This water changed to steam used for air stripping from the reactor. The temperature in preheater kept at 400°C. After stripping air from the reactor, the flow of water was stopped and began to n-heptane flow at a rate 4.5 ml/min to the reactor for ten minute period. The reaction products were flown through double pipe heat exchanger, internal tube ice bath, separation and collection for liquid storage, while the gaseous products passed through the liquid products and were collected in a vessel over water. The gas volume is determined by water displacement. A sample of gaseous product was collected and then analyzed by gas chromatography device (GC) (Dani, Model GC 1000 DPC), to determine the chemical composition of the gaseous product results as shown in **Table 1**. Cracking liquid products also collected in a tube and then analyzed by gas chromatography, to determine the chemical composition of the liquids product. The catalyst was replaced after the end of each experiment.

Table 1: n-heptane catalytic cracking using prepared zeolite catalysts

Catalyst	Temp., °C	Substances (wt%)														
		Methane	Ethane	Propane	Butane	Pentane	Ethene	Propylene	1-Butene	2-Butene	1-Pentene	2-Pentene	1-Hexene	2-Hexene	3-Hexene	n-Heptane (unreacted)
HY-Zeolite	400	7.77	–	–	26.1	–	–	31.9	–	6.57	–	–	6.19	4.41	–	17.1
	425	7.87	–	–	24.2	–	–	32.8	–	6.65	–	–	6.29	4.44	–	17.8
	450	7.96	–	–	22.2	–	–	33.8	–	6.72	–	–	6.39	4.48	–	18.5
	475	8.06	–	–	20.3	–	–	34.7	–	6.79	–	–	6.48	4.51	–	19.2
	500	8.15	–	–	18.3	–	–	35.5	–	6.86	–	–	6.57	4.55	–	20.1
	525	8.24	–	–	16.4	–	–	36.5	–	6.93	–	–	6.66	4.58	–	20.7
	550	8.33	–	–	14.5	–	–	37.4	–	7.00	–	–	6.75	4.62	–	21.4
Rh/HY-Zeolite	400	0.35	3.25	1.48	14.3	0.75	0.15	23.7	17.2	16.7	0.15	0.11	–	–	–	21.9
	425	0.41	3.39	1.55	15.1	0.78	0.11	25.4	17.4	17.3	0.11	0.12	–	–	–	18.3
	450	0.36	3.45	1.49	15.8	0.73	0.18	27.2	16.2	17.5	0.16	0.12	–	–	–	16.8
	475	0.36	3.40	1.54	16.4	0.74	0.12	28.6	16.3	17.9	0.16	0.16	–	–	–	14.3
	500	0.35	3.33	1.58	17.3	0.71	0.15	24.4	16.4	18.1	0.15	0.11	–	–	–	17.4
	525	0.33	3.75	1.56	17.9	0.74	0.10	25.9	17.2	18.4	0.13	0.15	–	–	–	13.8
	550	0.32	3.05	1.42	18.9	0.75	0.13	28.7	17.5	19.1	0.12	0.13	–	–	–	9.88

VII. CONCLUSION

1. BP showed a good ability to remove rhodium from SSAS using fixed bed adsorption unit. So, it could be recommended for removal of rhodium from wastewater instead of other material because it is valid, cheaper, economical, easy and simplicity for using, and has a high ability to adsorb rhodium, can be used several times by costly regeneration method and can be used finally in another benefit use.
2. Maximum removal of rhodium was 96.15% at initial rhodium concentration of 1 mg/l.
3. The percentage removal of rhodium was increased with decreasing flow rate of SSAS, and initial concentration of rhodium while the percentage removal was decreased with increasing of pH, treatment time and the height of adsorbent material BP.

4. It can be prepared a catalyst promoter for cracking process of n-heptane from the residual samples of BP that adsorb rhodium from aqueous solution to produce a fuel of higher octane number and remove the hazards radioactive waste in economic and eco-friendly method.

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