RECOVERY OF HEAVY METALS FROM SPENT CATALYST USING CHELATION TECHNOLOGY

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by

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DEPARTMENT OF CHEMICAL ENGINEERING

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Dedicated To My Father-In-Law

LATE MR. MADAN GOPAL SINGH

CERTIFICATE

This is to certify that the thesis entitled **“Recovery Of Heavy Metals From Spent Catalyst Using Chelation Technology”,** being submitted by **Ms. Garima Chauhan** to the **Indian Institute of Technology Delhi**, New Delhi, for the award of the degree of **Doctor of Philosophy** in Chemical Engineering, is a record of bonafide research work carried out by her. Ms. Garima Chauhan has worked under my guidance and supervision and has fulfilled the requirements for the submission of the thesis.

The research report and results presented in this thesis have not been submitted in part or in full to any other university or institute for the award of any degree or diploma.

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Garima Chauhan

Abstract

Green chemical engineering perceives the concept of developing innovative environmental benign technologies and improving the resource intensity. In order to explore this concept for minimizing the industrial waste and for reducing the environmental impact of hazardous chemicals, efforts have been made in present study to look for green approach to extract metals from spent catalyst which contain substantial quantity of heavy metals and may affect the ecosystem.

The present study explores the concept of chelation technology to develop an ecofriendly process for extraction of heavy metals from spent catalyst obtained from refinery and fertilizer industry. Ethylenediaminetetraacetic acid (EDTA) was employed for extraction of nickel (Ni) from spent catalyst. A biodegradable chelating agent [S,S]-ethylenediaminedisuccinic acid (EDDS) was also looked into as an environmental benign substitute of EDTA under atmospheric reflux and autogenous reaction conditions. Transport of metal−chelate complex and dissolution of target metals in aqueous medium were investigated by varying different process parameters (molar ratio of chelating agent to metal (MR), solid to liquid ratio (S/L), reaction time, reaction temperature, pH, stirring speed, particle size). Milder reaction conditions and high extraction efficiency, in addition to the added advantage of biodegradability makes EDDS competitive for sustainable technology development.

Simultaneous extraction of heavy metals from multimetallic spent catalysts using chelation technology was performed first time in the present study. Possibility of internal and external mass transfer resistance was investigated to improve the diffusion rate of reactants, while kinetic aspects were studied to achieve thermodynamic equilibrium for the process. Extraction of 80.4% Co and 84.9% Mo was achieved at optimum reaction conditions. Efforts were also made to recycle the recovered metals (Ni, Co, Mo) for the preparation of new fresh catalyst. Structural analysis of spent catalyst, recovered support material, and synthesized catalyst from extracted metals suggested successful recovery and recycling of metals. An innovative combination of chelation-dechelation concept was commenced to recover chelating agent and 98% chelating agent was precipitated. It was recycled upto four cycles with significant extraction efficiency (72.7% Co and 76.5% Mo) which manifests the ecology and economic sustainability of the process.

In parallel with the experimental investigations, analysis of structural and thermodynamics properties of metal–ligand complexes using density functional theory provided an insight into mechanism of chelation process. Theoretical prediction of stability constants of various metal-ligand complexes was performed to estimate the degradation behaviour of ligands. Degradation pathways were deduced to examine the environmental impact of the proposed approach.

Applicability of response surface methodology (RSM) was explored in order to provide elaborated quality of information and to overcome the limitations of OVAT. A Box-Behnken design (BBD) was coupled with RSM to optimize the process parameters using statistical analysis. It turned out that metal extraction depends predominantly on MR, S/L and reaction time.

A theoretical generalized framework was also proposed for metal extraction process using chelation technology. Shrinking core model was employed to evaluate the controlling mechanism for the process under consideration. Different kinetic models were analyzed to choose the optimum one regarding their accuracy of fitting the experimental data obtained at different operating conditions for metal extraction process. Theoretical design of a pilot plant for processing of 1 tonne spent catalyst using chelation technology was also proposed.

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Nomenclature

k_1 = peleg rate constant ,

k_2 = peleg capacity constant,

β = extraction rate constant, ($E_1 \times \exp(E_0/E_1)$), $\alpha = 1/E_1$,

D = scale parameter, related to reciprocal of the extraction rate constant,

m = shape parameter ($m=1$, curve is characterized by exponential, if $m<1$, the curve is parabolic with a high initial slope followed by an exponential shape),

a_0 = the extraction yield recovered instantaneously

a_1 = diffusion rate constant.

F = Feed Size (F80)(6000 microns)

Wi = Standard work Index (16.1)

K = Constant, (200 for ball mills and 300 for rod mills)

Cs = % of Critical Speed (70%)

S = specific gravity of feed (S =1.4)

D_1 = Diameter of ball mill (1m)

Mi = Top ball size in the ball mill (inch)

g = gravitations acceleration (m/s^2)

R = radius of the ball mil. (m)

r = radius of the top ball size (m)

N_c = Critical speed of the ball mill (rpm)

G = net mass of undersize material produced per revolution (g)

T = total amount of undersize material obtained per revolution (g)

U = (%) of undersize particles in the feed

W = energy required to grind 1 tonne of feed material to the product (kWh)

B = Equivalent energy consumption per revolution (kWh)

a = Correction Factor for Diameter Inside Shell Lining

b = Correction Factor to include effect of 0% loading and mill type

c = Correction Factor for speed of mill

L = Length in feet of grinding chamber (m)

C_1 = Correction for cyclone feed concentration ($C_1 = (53\text{-}V/53)^{-1.43}$)

C_2 = Correction for pressure drop ($C_2 = (3.28 * ((\Delta P)^{-0.28}))$)

C_3 = Correction for Specific Gravity ($C_3 = (1.65/(G_s\text{-}G_l))^{0.5}$)

D_2= cyclone diameter

T_1 = Hot Fluid Inlet Temperature (140°C)

T_2 = Hot Fluid Outlet Temperature (50°C)

t_1 = Cold Fluid Inlet Temperature (35°C)

t_2 = Cold Fluid Outlet Temperature (50°C)

V_P = volume of precipitator unit when 80% tank is filled with slurry (m^3)

H_1 = height of the cylindrical section of dechelator unit (m)

R_P = radius of the dechelator (precipitator) tank (m)

H_2 = height of the conical section of the dechelator unit (m)