EXTRACTION OF NICKEL FROM FLY ASH OF HEAVY OIL USING AMMONIUM HYDROXIDE

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ABSTRACT

An experimental investigation to recover the nickel from ash of residue of fire power station using ammonium hydroxide was conducted. The experiments were conducted in 0.6 L glass baffled reactor with multi speed mixer. The effect of different rotational velocities studied (200, 400, 600, 800 and 1000 rpm) and different temperatures were (50, 60, 70 and 80)ºC and it was found that the recovery of nickel increased with increasing rotational velocity till the 600 rpm while there is no change in nickel recovery with increasing temperature till 70 ºC. Controlling steps were investigation and it was found that the transfer through the film surrounding the particle (interphase) is the controlling step.

KEYWORDS: **Nickel, Fly ash, Ammonium Hydroxide, extraction**

استخلاص النيكل من الرماد المتطاير من الوقود الثقيل باستخدام هيدروكسيد الامونيوم

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الموجز

تضمن هذا البحث دراسة امكانية استخلاص النيكل من مخلفات حرق الوقود الثقيل المستخدم في انتاج الطاقة الكهربائية بأستخدام محلول هيدروكسيد الامونيوم حيث تم اجراء مجموعة من التجارب عن طريق استخدام مفاعل زجاجي حجمة ٦٠٠ مللتر مزود بخلاط ذا سرع مختلفة حيث تم استخدام ٢٠٠، ٤٠٠، ٦٠٠، ٨٠٠ و ١٠٠٠ دورة في الدقيقة و كذلك تم اجراء مجموعة من التجارب باستخدام درجات حرارة مختلفة ٥٠، ٦٠، ٧٠ و ٨٠ درجة سيليزية وقد بينت النتائج امكانية استرداد النيكل و بنسب عالية ، كما تم دراسة الخطوة المحددة للانتاج و ظهر بانها انتقال المادة من الطور البيني المحيط بالمادة الصلبة.

INTRODUCTION:

Nickel is an important alloying metal used in all branches of metal industry. It imparts to alloys toughness, strength, and lightness, and anti-corrosion, electrical and thermal qualities. The weight percent of nickel in the oil-fired fly ash, about 1-2%, so it is a considerable source compared with that of natural minerals sources, and due to this reason international attention was conducted to recover the nickel from this source earlier (**Tsai, 1998)** .The processing of nickel recovery depending on the characteristics of the burned oil and the place of ash formation. Recovery of nickel from fly ash can be achieved by acid using 30 % H_2SO_4, or water leaching (**Stas, 2007**).

(**Tsai, 1998**) discussed the experimental extraction of vanadium and nickel from oil-fired fly ash. The results indicated that leaching of oil fired fly ash in 0.5 N of sulfuric acid lead to extracted 60% of Ni. When leached in 2 N sodium hydroxide solutions the extraction of nickel was negligible. (**Othman,1999**) studied the recovery of metals from spent catalyst using sulfuric acid and selective precipitation of nickel from other metals by an addition of sodium hydroxide. It was found that sulfuric acid as a leaching agent resulted significantly higher nickel concentration compared to nitric and hydrochloric acids, sodium hydroxide was used to adjust the pH of the solution. At the best pH, more than 80% of nickel recovery was obtained. After some preliminary tests, (**Stas, 2007**) used two leaching stages of fly ash to recover vanadium, molybdenum and nickel. First stage was an alkaline leaching of fly ash to recover vanadium and molybdenum followed by a second stage using sulfuric acid leaching of the residual ash to recover nickel. (**Emad, 2007**) studied the extraction of nickel using the hydrometallurgy method. The author recovered nickel from the residues using sulfuric acid and the result obtained showed the possibility of obtaining the highest recovery at acid concentration 3 M, temperature=100 C, time of leaching 90 min and S/L ratio 5 gm/L. It can be understood from above recovery methods that hydrometallurgical methods are widely used. Procedures such as solvent extraction, ion exchange, and crystallization are employed to recover rare metals such as nickel. Leaching is concerned with the extraction of a soluble constituent from a solid by means of a solvent. The process may be used either for the production of a concentrated solution of a valuable solid material, or in order to remove an insoluble solid, such as pigment from a soluble material with which it is contaminated by the proportion of soluble constituent, present, its distribution throughout the solid, the nature of the solid and the particle size (**Coulson, 2002**). In the leaching of soluble materials from inside a particle by a solvent, a general steps can occur in the overall process. The solvent must be transferred from the bulk solvent solution to the surface of the solid. Next, the solvent must penetrate or diffuse into the solid. The solute dissolves into the solvent. The solute then diffuses through the solid solvent mixture to the surface of the particle. Finally, the solute is transferred to the bulk solution. Many different phenomena encountered make it almost impracticable or impossible to apply any one theory to the leaching action (**Geankoplis, 1993**).

Factors influencing the rate of extraction are particle size, which is influences the extraction rate in a number of ways. Solvent, the liquid chosen should be a good selective solvent and its viscosity should be sufficiently low. Temperature, in most cases, the solubility of the material which is being extracted will increase with temperature to give a higher rate of extraction. Agitation of the fluid, it is important because this increases the divergence diffusion and therefore the transfer of material from the surface of the particles to the bulk of the solution (**Coulson, 2002**). The mass transfer coefficient determines the rate of mass transfer across a medium in response to a concentration gradient. There are two main mechanisms depending on whether the flow is laminar or turbulent (**Grisafi, 1998**). Equation (1) is used for the mass transfer determination (**Coulson, 2002**):

$$\frac{dM}{dt} = \frac{KA}{b}(Cs - C) \tag{1}$$

Where:

M mass of solute transferred in time t, t time, A is the area of the liquid-solid interface, b is the effective thickness of the liquid film surrounding the particles, K diffusion coefficient, C_s is the concentration of the saturated solution in contact with the particles, C is the concentration of the solute in the bulk of the solution at time t (**Coulson, 2002**).

The presence or absence of turbulence can be correlated with impeller. (**Geankoplis, 1993**) defined Reynolds number N_{Re}, as:

$$N_{Re} = \frac{D_a^2 N \rho}{\mu} \tag{2}$$

Where:

D_a is the impeller (agitator) diameter in m, N is rotational speed in rev/s, ρ is fluid density in Kg/m^3, and μ is the viscosity in Kg/m.s.
Many previous researchers used acidic leaching to recover metals, such as V, Ni, Fe, ---- etc. from waste ash, all of them are neglected the effects of corrosion on equipments. The aim of this work is to study the best conditions and then find the controlling step to nickel recovery from fly ash of produced from heavy oil-fired electrical power station due to a limited source of nickel in Iraq and to overcome the problem of corrosion.

EXPERIMENTAL WORK

Fly ash were collected from boilers of Al-Dora thermal power station in south of Baghdad. Prior to leaching, a mass of ash was ground and mixed for 12 h. in laboratory ball mill, then the sample was analyzed for nickel content by atomic absorption spectrometry and the average percentage was found to be 1.51wt % .Ammonium hydroxide NH_4OH (1N) was used as leaching agent , which is prepared from (25 %) NH_3 supplied by Scharlau.

EXPERIMENTAL APPARATUS

First of all the characteristics of reactor was studied carefully and the design was implementation. Experiments were carried out in (600 ml.) glass baffled reactor, the diameter of glass reactor was 9.5 cm and height of 12 cm with three rectangular baffles of 0.8 cm thickness, and 1.5 cm width. To insure a homogeneous distribution of temperature water bath with controller of temperature to heat the solution to the required temperature (50-80 ºC) was used. The reactor was supplied by a mixer consisted of stainless steel shaft, screwed with three blades impeller, the diameter of the impeller was 3 cm. the stirrer rotated by means of an electrical motor to achieve the required velocity. The mixer was screwed with stand and two hands to infixing mixer and thermometer (with range 0-250 ºC). The motor of mixer and water bath was connected to controller. In each experiment 80 g of ash was added to 400 ml. of 1 N ammonia solution and heated using water bath with different temperatures and stirred with different speeds (rpm), after one hour the solution was filtered and analyzed for nickel content by means of atomic absorption spectrometry. **Figure 1** shows the schematic diagram of the experimental apparatus.

RESULTS AND DISCUSSION:

Experiments were carried out for 1 h. at 80 ºC with different agitation speed (200,400, 600, 800, and 1000) rpm to study the effect of agitation speed on nickel recovery. **Figure 2** showed that the recovery of nickel depend on the agitation speed, 1.1 g/L was recovered at 400 rpm and increased with increasing agitation speed, for 600 rpm the recovery was 2.86 g/L, so the best value of recovery is at 600 rpm. Above this speed the recovery doesn't increase further. This result indicates that the dominant resistance of the leaching process is the mass transfer from the particles surface to the surrounding medium.
Agitation is important because it increases the eddy diffusion and therefore increases the transfer of material from the surface of the particles to the bulk of the solution (**Coulson, 2002**).
Figure 3 shows the effects of Reynolds number on the mass transfer coefficient. It is clearly that the mass transfer coefficient increased with increased Reynolds number, this can attributed to as turbulent flow is involved, and the turbulent-eddies act as a transport mechanism giving rise to transport rates that may be orders of magnitude higher than those due to molecular effects (**Grisafi, 1998**). The effect of temperature on nickel recovery was studied; experiments were conducted for 1 h, and 600 rpm, S/L ratio 1/5 and for different temperatures (50, 60, 70, and 80) ºC. figure 4 showed that the nickel recovery is not affected by temperature till 70 C and then a shift in nickel yield was occur between 70 ºC to 80 ºC, one can conclude that the controlling step of this process may changed from mass transfer control to kinetic control because according to the Arrhenius law the rate of reaction is increased with temperature of the process. If the intra-particle diffusion or kinetic of the process is the dominant resistance, the nickel recovery should be depends on the temperature,

the intra-particle diffusion is influenced by temperature, because it affects on the diffusion coefficients (**Leavenspile, 1999**) and (**Luis, 2000**).
Figure 4 shows the comparison of this work with (**Stas, 2007**) at different temperatures. In this work 94.7 % of nickel was recovered when using 1 M NH_4OH, (S/L) ratio =1/5, temperature= 80ºC after 1h. While (**Stas, 2007**) recovered about 76% of nickel using $[H_2SO_4]$ =5M, (S/L) =1/5, temperature 80ºC after 4h.

CONCLUSIONS:

1. The resedue of fired power station is a good source of nickel.
2. The recovery of nickel from ash via NH_4OH was successful conducter.
3. The best agitation speed for this process is 600 rpm.
4. A controlling step of this process was changed from mass transfer controlling step to kinetic controlling step.

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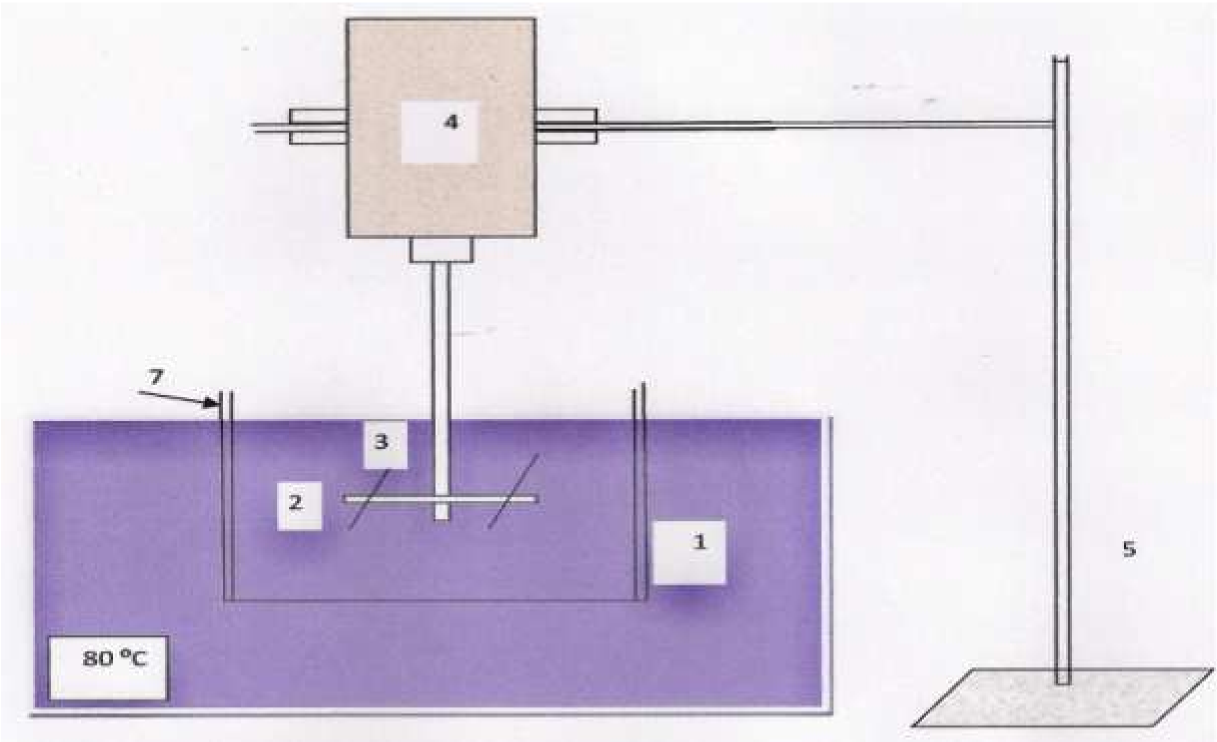

Figure 1 A Schematic diagram of the experimental apparats
1-Glass reactor, 2- Impeller, 3- Shaft, 4- Motor, 5- Stand, 6- Water bath, 7-baffle

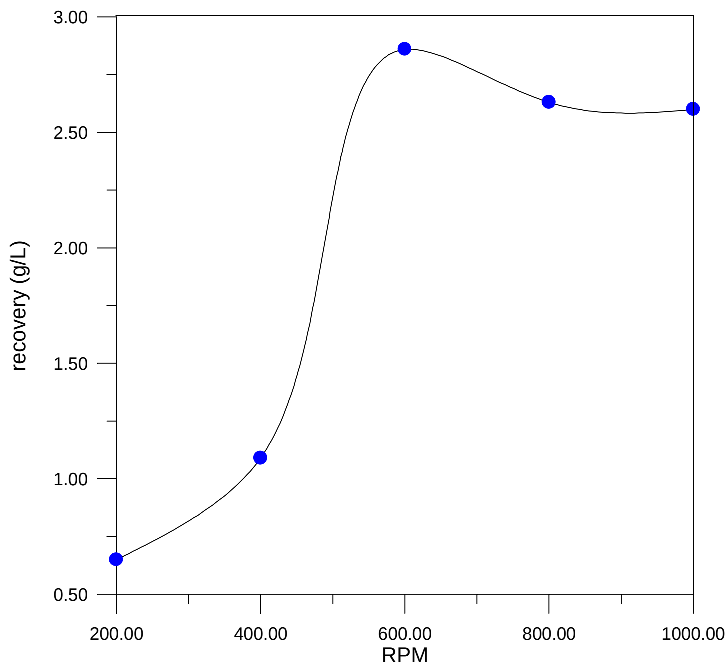

Figure 2 effect of agitation speed on nickel recovery at T=80 ºC

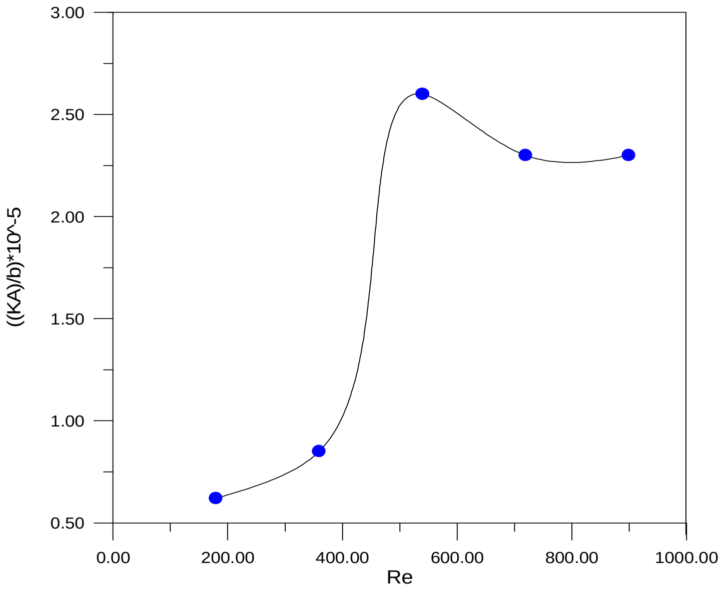

Figure 3 Effect of Re number on mass transfer coefficient

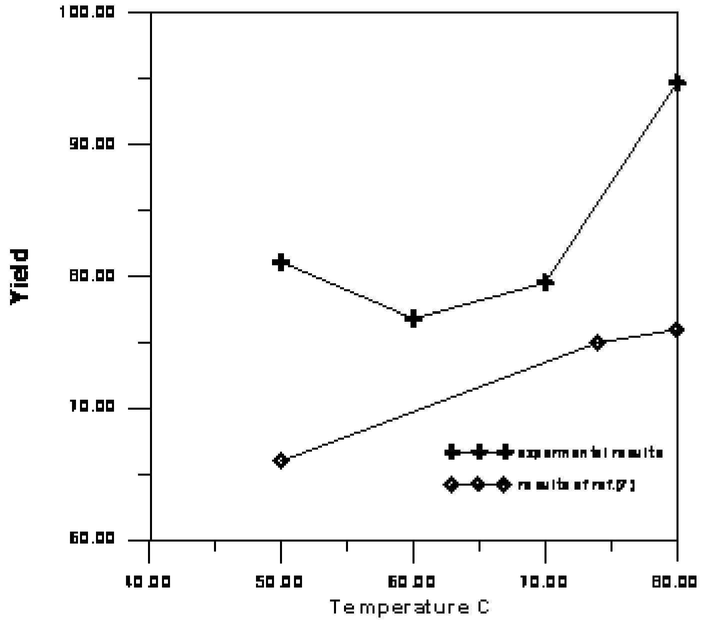

Figure 4 Comparison with results of (**Stas, 2007**)