Rwcovry of Nickel from Burned Fuel Waste in seawage station

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Abstract:

Best chemical conditions for the recovery of nickel as sulphate or oxide from burned fuel waste in seawage stations had been considered. The effects of different factors like acid concentration, acid volume, time of reaction, and pH on the recovery of nickel from the waste have been studied. It was found that the percentage of nickel in the waste (3 - 6) % and becomes (8 - 15) % ater the recovery of vanadium. The performance of nickel recovery is 90% and the purity around 98%.

Introduction:

Nickel is found as a constituent in most meteorites and often serves as one of the criteria for distinguishing a meteorite from other minerals. Nickel is obtained commercially from pentlandite and pyrrhotite. Nickel is silvery white and takes on a high polish. It is hard, malleable, ductile, somewhat ferromagnetic, and a fair conductor of heat and electricity. It belongs to the iron-cobalt group of metals and is chiefly valuable for the alloys it forms⁽¹⁾. Nickel and magnesium can be recovered in high yields and virtually free from vanadium and each other from natural bitumen ash. The separation of nickel from magnesium for metals recovery can thus be done without interference from high levels of vanadium. For purpose of recovering the nickel, the ion exchange procedure commonly used in the prior art is suitable and the magnesium may be recovered by precipitating the carbonate or hydroxide⁽²⁾.

The combustion of heavy fuel oil power generation leads to great production of fly ashes, usually disposed off in controlled expensive landfills.

Hydrometallurgical processes are used for the recovery of valuable metals such as vanadium and nickel carbonaceous residues, so obtained, could be burned to recover energy⁽³⁾. Vanadium and nickel values can be recovered from petroleum residue by a simple and economical process using inexpensive reagents and available equipment⁽⁴⁾.

Nickel occurs in nature mainly in combination with arsenic, antimony, and sulphur, for example, as millerite, (NiS), as a red Ni ore that is mainly NiAs, and in deposits containing chiefly ofNiSb, NiAs2, NiAsS, orNiSbS⁽⁵⁾.

In this work, nickel is extracted from the solid waste (after extracting vanadium) as nickel sulphate where nickel oxide was prepared thereater (from the sulphate)⁽⁶⁾.

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Experimental:

Apparatus:

Heating mantel, beakers, mechanical stirrer, Buckner funnel, chiller (water cooler), and vacuum pump.

Chemicals:

Sulfuric acid, Ammonium hydroxide, sodium sulphate, burned fuel waste.

Procedure:

- 7.5 liter of (4 M) sulfuic acid was added to 1 kg of the solid waste after extracting vanadium from it.
- Heating the solution to 100 °C with stirring for 4 hours.
- After cooling, the solution was filtered and the precipitate which consists about (20 30) % of the solid waste was discarded.
- **Root** (A):

700 ml of ammonium hydroxide for each litre of filtrate was added with continuous cooling at10°C. The solution was left3 hours for nickel precipitation as ammonium salt of nickel disulfate [NiSO₄.(NH₄)₂SO₄.6H2O]. N.B: This root was used in this work because of its low cost.

Root (B):

Another method for precipitating nickel is by the addition of 20 gm of sodium thiosulfate (Na₂S₂O₃) for each liter of filtrate with heating for half an hour to dissolve the thiosulfate. Ammonium hydroxide was added with stirring and cooling at (10 °C) till the pH reaches the value of (1); (the thiosulfate was added to mask the precipitation of vanadium with nickel when pH=l)⁽⁷⁾.

• The solution was filtered and the filtrate was discarded. One kilogram of the precipitate was dissolved in 5 L of H₂O, and then to this was added NH₄OH solution till the pH becomes equal to 9. The solution was heated to boiling with continuous stirring where most of the nickel has been dissolved. The solution was filtered and the precipitate discarded (which contains small amounts of vanadium, iron, and nickel). The volume of the filtrate was reduced by evaporation to the smallest volume and the pH was balanced at the value of 2 for precipitating nickel as [NiSO₄.(NH₄)₂SO₄.6H₂O] which looses its water of crystallization at 300 °C, and when the temperature reaches 700 °C the compound changed to NiSO₄.

Results and Discussion:

The highest percent of recovery was about (98) % as shown in table (1) which shows the concentration of sulfuric acid (2 - 6) M where the recovery between (33 - 98) %. In our study we used (4 M H₂SO₄) which is the best concentration and avoiding highest concentrations of the acid and to decrease the impurities in the recovered solution. In order to get the best volume of acid, different volumes where used (50 - 150) ml for each 10 gm of solid waste and the recovery was between (80 - 96) percent as shown in table (2). It can be noticed that the percent

of recovery by acid volumes (75, 100, and 150) ml were close to each other. To let the process economical, 75 ml of acid/ 10 gm of solid waste were used taking into account the large volumes needed in the production stage.

The time also was taken as one of the factors that will influence the recovery of nickel and the data were shown in table (3) where the percent of recovery (68 - 98) and the chosen time were 3 hours. Table (4) show the effect of pH on the precipitation process where the percent of impurities increased when the pH was increased. It was found that the best pH for the precipitation (1 - 2.5) and also best precipitation was found at higher acid concentrations (pH = 1).

In order to and the minimum amount of ammonium hydroxide that gives the highest amount of precipitate, different volumes of NH_4OH were used as shown in table (5). It was shown that as the amount of NH_4OH increased, the amount of the precipitate will increase and the co-precipitation of the impurities will increase too. The minimum volume of ammonium hydroxide was found to be equal to 70 ml.

The presence of vanadium in solution made the precipitation process more complex since it will co-precipitate with nickel. To get rid of this phenomenon, the pentavalent vanadium was reduced by sodium thiosulfate $(Na_2S_2O_3)$ as shown in table (6) where 1 gm of the thiosulfate for each 100 ml of the recovery solution was enough.

The time of precipitation was also studied where 90 % of precipitation was achieved after 6 hours from the time of the addition of the base as shown in table (7). Table (8) shows the comparison between the precipitation data and the purity of the precipitate (the amount of vanadium and iron that will co-precipitate with nickel) and the percent of precipitation with and without the use of the thiosulfate. Higher percent of precipitation with low percent of impurities was found when using thiosulfate at (pH = 2) compared with that at the same pH without the use of the thiosulfate. Table (9) shows the analysis data for the recovery of nickel sulphate.

Table (1): Shows the percent of recovery of nickel as a function of acid concentration Weight of waste sample (10) gm, Time of heating (4) hours ,Temperature 100 °C, Volume of acid (100) ml

| Cone, of Acid | Quantity of Ni | Quantity of Ni | %N |
|---------------|----------------|----------------|-----------|
| (mole/ litre) | in soln. (gm) | in sample (gm) | Recovered |
| 2 | 0.33 | 0.98 | 33.7 |
| 3 | 0.72 | 0.98 | 73.5 |
| 4 | 0.93 | 0.98 | 94.9 |
| 5 | 0.93 | 0.98 | 94.9 |
| 6 | 0.97 | 0.98 | 98.9 |

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Table (2): Shows the percent of recovery of nickel as a function of acid volume Weight of waste sample (10) gm, Time of heating (4) hours, Temperature 100 $^{\circ}$ C, Concentration of acid (4) M

| Cone, of Acid | Quantity of Ni | Quantity of Ni | %N |
|---------------|----------------|----------------|-----------|
| (mole/ litre) | in soln. (gm) | in sample (gm) | Recovered |
| 50 | 0.991 | 1.227 | 80.8 |
| 75 | 1.158 | 1.227 | 94.4 |
| 100 | 1.154 | 1.227 | 94.05 |
| 150 | 1.177 | 1.227 | 95.9 |
| 50 | 0.991 | 1.227 | 80.8 |

Table (3): Shows the percent of recovery of nickel as a function of time Weight of waste sample (10) gm, Concentration of acid (4) M, Temperature 100 °C, Volume of acid (100) ml

| Heating time (hour) | Quantity of Ni in soln. (gm) | Quantity of Ni in sample (gm) | %Ni Recovered |
|------------------------|---------------------------------|----------------------------------|------------------|
| 1 | 0.629 | 0.918 | 68.5 |
| 2 | 0722 | 09 | 8 |
| 3 | 0898 | 09 | 8 |
| 4 | 090 | 0918 | 980 |

Table (4): Shows the precipitation of nickel as a function of pH Weight of waste sample (10) gm, Weight of thiosulfate (2) gm, Time of precipitation (20) hours, percent of Ni in sample (11.68) %

| рН | %Ni | % V | %Fe |
|-----|-------|------|------|
| <1 | 11.67 | 0.31 | 0.15 |
| 1 | 11.66 | 0.45 | 0.2 |
| 1.5 | 1 | .66 | 0.44 |
| 2.0 | 1 | 8 | 0.48 |
| 2.5 | 11.12 | 0.53 | 0.41 |
| 3.0 | 6.13 | 2.70 | 4.66 |
| 4.0 | 5.86 | 2.91 | 4.86 |

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استرداد النيكل من مخلفات حرق الوقود في المحطات مياه الصرف الصحي أحمد جاسم مخلف الجامعة التكنولوجية / قسم العلوم التطبيقية / فرع الكيمياء التطبيقية

الخلاصة :

تضمن البحث أفضل الطرق الكيميائية والظروف المناسبة لاسترداد النيكل على شكل كبريتات أو اوكسيد من مخلفات حرق الوقود في محطات مياه الصرف الصحي تم دراسة بعض المتغيرات مثل تركيز الحامض المستخدم ، حجم الحامض ، زمن التفاعل والأس الهيدروجيني (pH) على عملية استرداد النيكل من المخلفات بعد استرداد الفناديوم منها . تشير النتائج المستحصلة بأن نسبة النيكل في المخلفات تتراوح بين (3-6) % وتصبح النسبة (8-15) % بعد عمليات استرداد الفناديوم منها . كانت كفاءة عملية استرداد النيكل اكثر من (90) % ونقاوة المادة الناتجة بحدود (98) % .

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