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Hydrothermal Leaching of Nickel, Uranium and The Remaining Vanadium from Alkali Treated Boiler Ash Residue Using Citric Acid

El-Sayed A. Manaa*, Mohamed N. Kouraim, Mohamed A. Youssef, Ahmed R. Bakry, Ekramy El-Desoky
Nuclear Materials Authority, Research Sector, El Maadi, Cairo, Egypt.

IN this study; the boiler ash was subjected to a digestion process using citric acid solution. Previously the ash was treated with $NaCO_3$-$NaHCO_3$ solution for vanadium leaching. In this step about 85% of the vanadium content was leached while nickel was not dissolved using the carbonate solution. The ash after vanadium dissolution was digested using citric acid for nickel and the remaining vanadium beside uranium dissolutions. Experimental results have shown that maximum metals dissolution obtained using 0.3M citric acid was 81, 85 and 89% respectively. The resultant liquor solution was subjected to successive nickel, vanadium and uranium separations. Firstly; the leach liquor is treated with dimethylglyoxime solution to precipitate Ni species. Secondly; the filtrate after Ni separation is treated with TBP/kerosene solvent for uranium extraction. Finally, the aqueous solution is treated with ammonium chloride to precipitate the vanadium content.

Keywords: Nickel, Vanadium, Uranium, Boiler ash, Citric acid.

Introduction

Nickel and vanadium metals/oxides are very essential for many industries mainly in coinage alloys, electroplating, alloys, aerospace and batteries in the nuclear applications [1]. Naturally, Ni and V are present in many ores but the recovery of Ni and V from their natural resources is not sufficient for their uses. Therefore, the world is interested by the ashes as additional and economic V and Ni resources [2-4]. For uranium, it a strategic element especially for nuclear applications in spite of its concentration in the earth crust is 4 ppm distributes in many ores. Internationally, huge amounts of ashes are generating from the oil-fired power stations and in Egypt, approximately 4000 metric tons of oil ashes generate yearly. Physically, the ash is divided into fine and coarse parts based on the particle size; the fine is the fly ash while the coarse is the boiler residue on the other hands; chemically the ash is composed of organic and inorganic portions. The organic portion; contain nitrogen compounds beside high carbon content while the inorganic

one that having heavy metals [5-7].Vanadium in ashes is 3-40% beside nickel is 1-13% while the uranium content is 7-46 ppm reach to 400 ppm according the ash nature, then ash is conceder as Ni and V source [8]. In order to leach V and Ni from ash, physical, pyrometallurgical and hydrometallurgical methods are used [9]. In spite of the physical techniques are simple and environment-friendly, but is considered as a pretreatment step. On the other hand, the emitted gases (*SO_2, dioxins, and furans*) beside the formed refractory V-Ni compounds limiting the pyrometallurgical methods using. The hydrometallurgical techniques using alkaline solutions are effective for V dissolution especially from Fe and Ni species but not from Si [8]. Using mineral acids for Ni and V dissolution in spite are not leach Si but have many disadvantages as; low selectivity besides high acid consumption [9]. To avoid the previous disadvantages, leaching processes using organic acids are performed [10-11]. Some works were applied for Ni and V dissolution using citric acid [12-13]. The

leaching of V, Ni, Mo and Co metals from spent hydro-treating catalysts using organic acids was studied [14]. The results indicated that oxalic acid showed the highest metals leaching. The leaching of Ni from laterites using organic acids indicated that the most effective acids were citric, lactic, gluconic, pyruvic and tartaric [15]. Using oxalic, citric and tartaric acids for V and Ni leaching from a hydro-processing catalyst's residue shown that the metals leaching efficiencies were the same but in the presence of $Fe(NO_3)_3$, $Al(NO_3)_3$ or H_2O_2 as oxidants [16-17]. The obtained results during Ni and V dissolutions from a spent catalyst using organic acids, water and aldehyde show that; the leaching efficiencies were 95 and 85% for V and Ni using citric or tartaric acids respectively [18]. The results obtained during Ni and V leaching from a catalyst ore using citric acid indicated that, the leaching efficiencies using citric were higher than by using H_2SO_4 [19]. Maximum Ni extraction from the spent refinery processing catalyst using citric acid obtained at 80°C after 10h [20]. The extraction of Ni using citric, sulfuric, hydrochloric, nitric, lactic and oxalic acids from low-grade saprolitic ores shown that citric was the best-used acid [14]. For uranium dissolution by organic acids many types of research were applied [21-22]. Generally, the alkaline solutions leach uranium but after an oxidation step because uranium in the ash after alkaline treatment with CO_3-HCO_3 solution presents as U(IV) [22]. The aim of this work is optimizing an environment, economic and effective method for Ni, V and U recovery from the ash residue using citric acid.

Materials and Methods

Sample specifications and Analytical procedures

The original sample was collected from El-Kriymat steam electric station, Cairo, Egypt. The boiler ash refers to the residue falls down through the air flow to the power stations bottom of the boiler and its content is around 12% from the generated ashes [23]. To avoid the pollutions as well as minimize landfill space, the ashes should be treating and reusing. In previous work, the bottom ash was beforehand treated with a CO_3-HCO_3 solution for vanadium leaching [22]. The remaining brown cake was then subjected to a hydrothermal digestion process using citric acid ($C_6H_8O_7$) for nickel and the remaining vanadium beside uranium dissolutions. In this work, the chemical reagents used were analytical grade. Nickel was determined spectrophotmetrically using an alcoholic dimethylglyoxime solution

(*DMG*) at pH 7[24]. Vanadium was estimated spectrophotmetrically using 2-(5-Bromo-2-Pyridylazo) 5-diethyl aminophenol dye [25]. Uranium was determined by the oxidimetric titration against ammonium metavanadate and ammonium ferrous sulfate using diphenylamine indicator [26]. Silica, alumina and titanium contents were determined spectrophotmetrically using molybdate, alizarin and tiron reagents respectively [27]. Sodium and potassium ions were determined photometrically using *JENWAY PFP7 (UK)* flame photometer [28]. Loss of ignition was evaluated thermally at 1000 °C (**Table 1**). On the other hand, trace elements were determined using *UNICAM 969* (*UK*) atomic absorption spectrophotometer.

Leaching procedures

The leaching experiments were performed in a 150 ml 2-neck glass flask equipped with water-cooled condenser to minimize the solution evaporation (**Figure 1**). The ash was mixed with 0.3M citric acid at acid/ash ratio of 10/1 (*ml/g*) at 85°C for 4 h stirring. The digested solution after cooling was filtrated then subjected to metals analysis. The observation shown that, the magnetic bar captured the main iron content as magnetic impurities while the remaining iron in the sample was slightly reacted with citric acid [29]. The metals dissolution efficiency was calculated using equation (1).

$$\alpha \ (\%) = (A-B)/A*100 \qquad \text{eq.1}$$

Where α is the metal leaching efficiency, *A* is the metal concentration (% or ppm) in the original sample and *B* is the metal concentration of remaining residue

Results and Discussions

For the studied metals dissolutions from the treated boiler ash; many factors were studied and optimized. Factors include; the ore fineness, citric acid concentration, citric acid/ore ratio, temperature, stirring speed beside digestion time.

Effect of ore fineness

Generally, the hydrothermal leaching is a grain surface dependent process. For study the effect of the ash fineness on Ni, V and U leaching using 0.5 M citric acid, four experiments were performed using particle size fractions ranged from -140 to -270 mesh size (105-53 μm). The experiments were studied at 85°C with citric acid/ash ratio of 10/1 for 4h leaching time at 400 rpm stirring speed. The results in **Figure 2** showed that the

dissolution rate of Ni, V and U increased as the ash fineness increased. This can be attributed to the increase in contact surface that, accompanies the decrease in particle size per the solid. This is due to the fact that; smaller particles have a large surface area available for carrying out metal's dissolution. The leaching efficiencies of Ni, V and U increased from 65.2, 75.3 and 72.2% to 81.9, 85.7 and 87.1 % as the sample fineness increased from 105 to 74 μm respectively. More sample finenesses to 53μm led to a slight Ni and V increase leaching efficiencies to 82.1 and 86.1% respectively while the uranium leaching efficiency remains as at 74 μm (86.8%). Therefore; economically

TABLE 1. Major oxides and trace elements content in the alkali treated boiler ash sample.

Major elements		Trace elements	
Element oxide	W%	Element	ppm
V_2O_5	**3.41**	Pb	1201.12
NiO	**13.26**	Cd	154.81
Na_2O	23.65	Sr	801.71
Fe_2O_3	14.83	Cu	921.52
MgO	7.72	Cr	304.11
Al_2O_3	4.63	Ga	91.26
SiO_2	2.91	Ba	70.21
K_2O	4.52	As	44.13
CaO	2.31	**U**	**13.94**
ZnO	1.33	Th	5.15
TiO_2	1.01	Ce	3.61
Loss on ignition (L.O.I)*	19.13	Gd	3.21
		Sc	0.48
Total (%)	**98.71**	Eu	0.35

*L.O.I: refers to moisture, adsorbed, combined water and organics
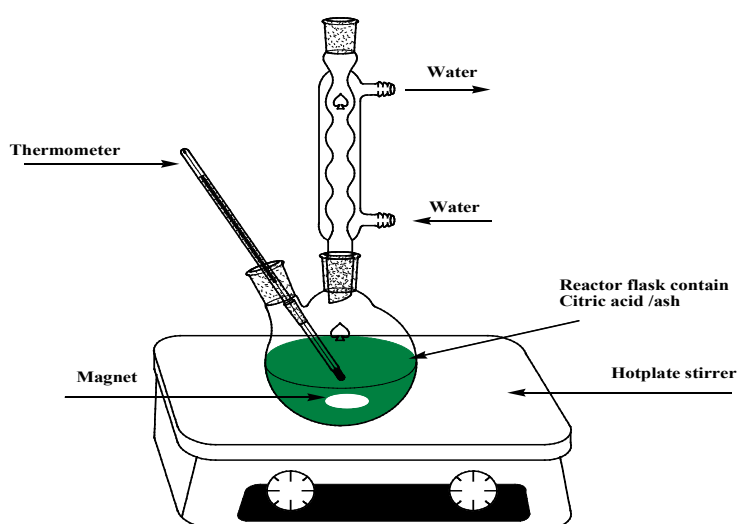

Fig. 1. Metals leaching procedures using citric acid from the treated boiler ash.
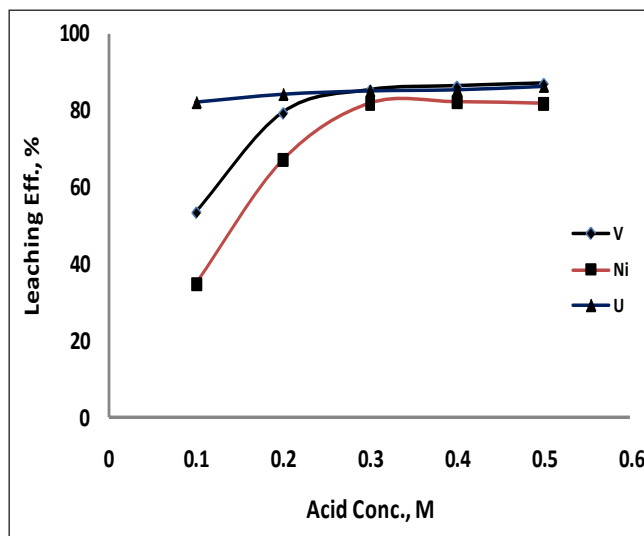
Fig. 2. Effect ash grain size (-mesh size) on Ni, V and U leaching using citric acid.

74μm (-230 mesh size) would be considered as the optimum sample fineness used for the studied metals dissolutions.

Effect of citric acid concentration

The effect of citric acid concentration upon Ni, V and U leaching from the ash (74 μm) was studied using different citric acid concentrations ranged from 0.1 to 0.5 M while the leaching temperature was fixed at 85°C for 4 h with S/L ratio of 1/10. The results in **Figure 3** demonstrated that; the citric acid concentration has a positive Ni and V dissolution effect but has a moderated uranium leaching effect. Nickel and vanadium leaching efficiencies were increased from 34.7 and 53.1 to 81.7 and 85.2% as the citric acid concentration increased from 0.1 to 0.3 M respectively while the uranium leaching was slightly increased from 82.2 to 85.1%. This effect is due to increase the hydronium ion (H_3O^+) with increase the citric acid content, which led to further metals dissolution. Increasing the citric acid concentration than 0.4M hasn't any significant metals leaching improvement. In general chelating agents leach metals by adsorbing on the ore surface and forming soluble complexes. The formed complexes at the mineral surface shift the electron density toward the metal ion, which destabilizes the Me-O lattice bonds and facilitates the metal dissolution [30].

Although citric acid is classified as a weak acid, it is an excellent leaching agent and metals binder. Citric acid leach metals via two proposed mechanisms, the first is direct displacement of the metal ion by hydrogen ions (*acidolysis*), while the second is formation soluble metal complexes which lead to the effectiveness of citric acid for metals leaching than mineral acids [31].

Commonly the weak organic acids as citric reduce the positive charge of metal cations then increase the metals dissolution efficiencies [32]. The nickel, vanadium and uranium leaching using citrate may be explained through several processes, including the complexation with citrate species. Uranium and nickel species react with citrate with 3:2 ratio while vanadium reacts with 2:2 ratio. The proposed chelating reactions of citric acid with Ni and V species were presented in Figure (4).

Effect of liquid/solid ratio

The effect of citric acid/ash ratio (ml/g) on leaching of the studied metals from the ash (74 μm) was studied using 2.5/1 to 12/1 ratios using 0.3M citric acid at 85°C for 4h digestion time under 400 rpm stirring speed. Data in **Figure (5)** maintained that, at acid/ore ratio less than 5/1 the citric acid as a leaching solution was insufficient for metals dissolution due to the sludge formation but Ni, V and U metals leaching efficiencies were increased from 11.5, 19.7 and 31.2 to 20.9, 35.8 and 56.3% as acid/sample ratio increased from 2.5/1 to 5/1 respectively. As the acid/ore ratio increased from 8/1 to 10/1, the leaching efficiencies of Ni and V were sharply increased from 41.8 and 65.7% to 82 and 85.1% while the uranium leaching efficiency was slightly increased from 83.1 to 87.2% respectively. Increasing the acid/ash ratio to 12/1 led to a slight V leaching increase (86.5%) while Ni leaching was reduced to 81.1%
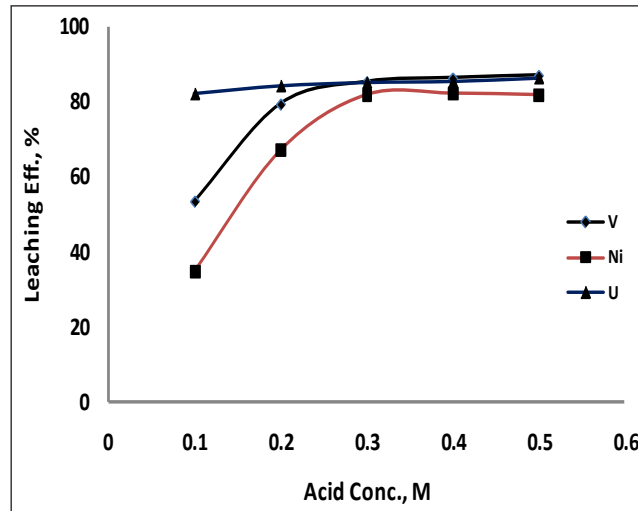
Fig. 3. Effect of citric acid concentration on Ni, V and U leaching from the ash residue.
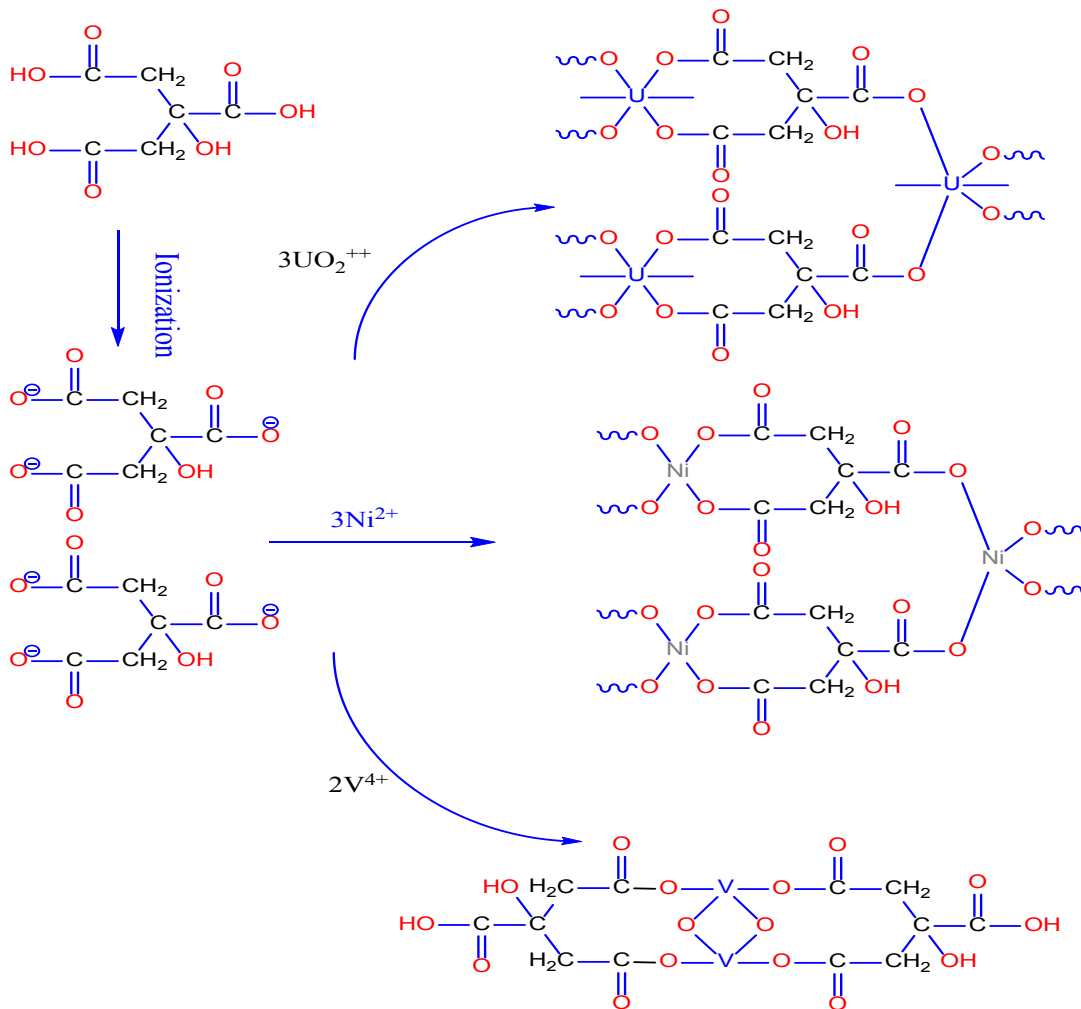

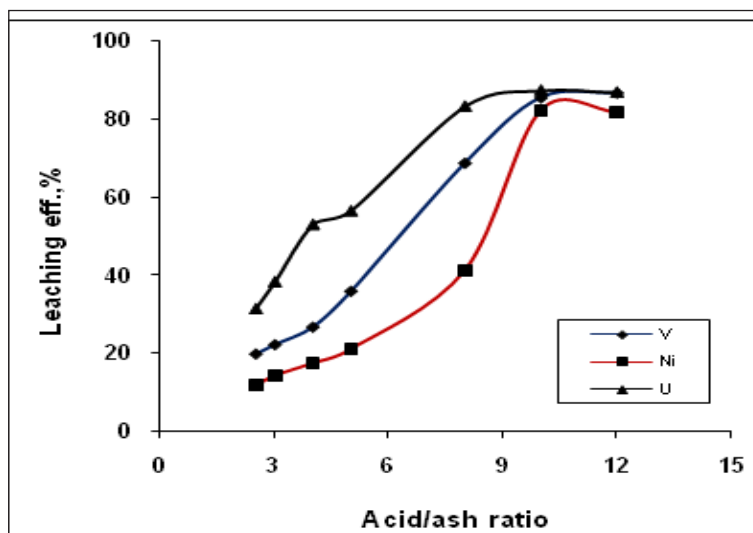**Fig. 4. Schematic reaction of citric acid with Ni, V and U species.**

Fig. 5. Effect of acid/ash ratio on Ni, V and U leaching from the ash residue using citric acid.

and the uranium leaching was nearly remained at 10/1 acid/ash ratio. The optimum citric ratio was chosen at 10/1 in all subsequent leaching tests.

Effect of digestion temperature

The effect of digestion temperature on Ni, V and U leaching from the fine ash (74 μm) using 0.3M citric acid was studied using acid/solid ratio of 10/1for 4h time under 400 rpm stirring speed at various temperatures from room temperature (28°C) to boiling (105°C).

In general, the metals leaching mechanisms using citric acid depend on the reaction temperature. The results in **Figure 6** indicated that; the leaching process is an endothermic reaction so increasing the temperature improved leaching of the studied metals. The dissolutions of Ni, V, and U at room temperature were 14.3, 32.1 and 54.7% respectively. The leaching efficiencies of Ni, V, and U metals increased to 82.2, 84.8 and 87.6 % by increase the reaction temperature from 28 to 85°C respectively. Rising the reaction temperature to boiling (108 °C) led to Ni and V leaching decrease to 73.1 and 78.3% respectively which might due to the partial ionizing of citric acid at higher temperatures. The uranium leaching efficiency was stabilized at 87%, so the optimum leaching temperature was chosen at 85°C. At room temperature, the reaction mechanism is a surface area-controlled process, but under higher temperature citric acid diffuses into the grain core thus split into fragments that accelerate the metals leaching rate [33].

Effect of stirring speed

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The stirring effect on Ni, V and U leaching from the treated ash (74 μm) using 0.3M citric acid was investigated at various stirring speeds from 50 to 500 rpm with acid/solid ratio of 10/1 at 85°C for ٤h digestion time. In general; increase the stirring speed usually increase the dissolution of the metal due to increasing the collection between the ore and the leachate (*sludge formation prevent*) besides decreasing the thickness of the mass transfer boundary layer on the surface of the particles. The results in **Figure 7** indicated that; at 50 rpm stirring speed, the ash particles subsiding in the flask bottom has occurred. The leaching rate of the studied metals nearly was in the same range at stirring speed 250 to 500 rpm. Therefore, the stirring speed was kept at 350-400 rpm in leaching experiments.

Effect of the leaching time

The effect of digestion time on Ni, V and U dissolutions from the treated ash (74 μm) using 0.3M citric acid was examined at various time intervals ranged from 15 to 300 min while the other leaching conditions were fixed at 10/1 acid/ash ratio at 85°C reaction temperature under stirring. The obtained results in **Figure 8** indicated that, as the contact time increased from 15 to 240 min, the leaching efficiencies of Ni, V and U were increased from 10.2, 25.6 and 18.2 % to 80.8, 84.5 and 87.1% respectively. Prolonged the time to 300 min led to a slightly Ni leaching decrease (77.6%) and V leaching increase (86.2%) while U leaching efficiency nearly remained at the same value (87.2%) as the time increased from 4 to 6 h. Therefore, 4h considered
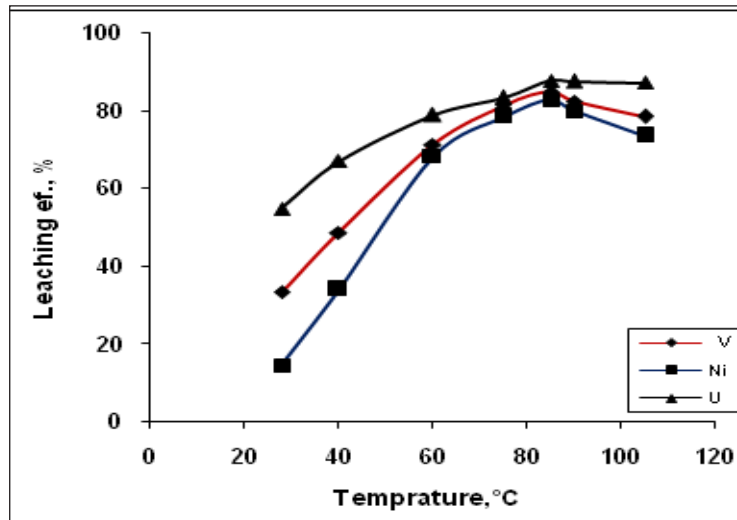
Fig.6. Effect of temperature on Ni, V and U dissolution from treated ash using citric acid.
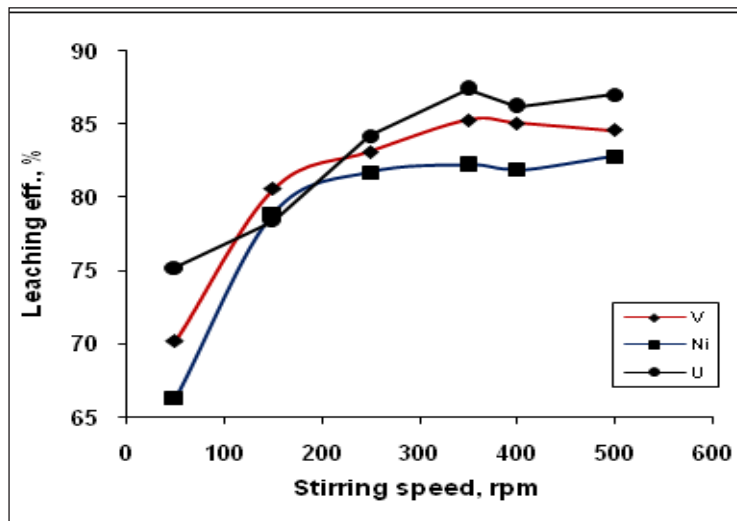

Fig.7. Effect of stirring speed on Ni, V and U leaching from treated boiler ash using citric acid.
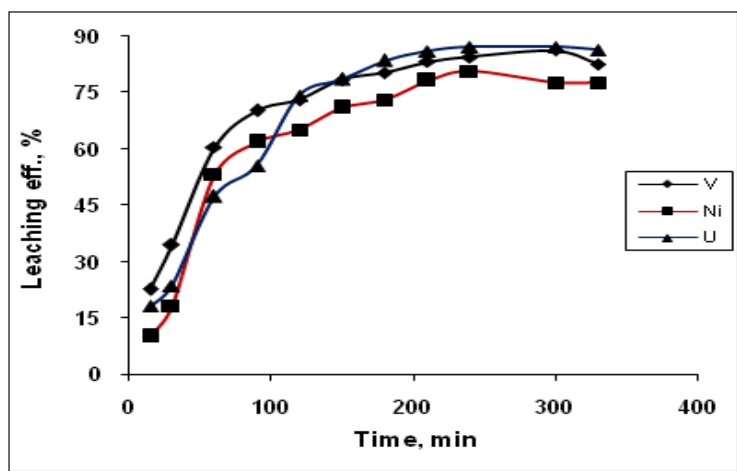

Fig.8. Effect of reaction time on Ni, V and U leaching from treated ash using citric acid.

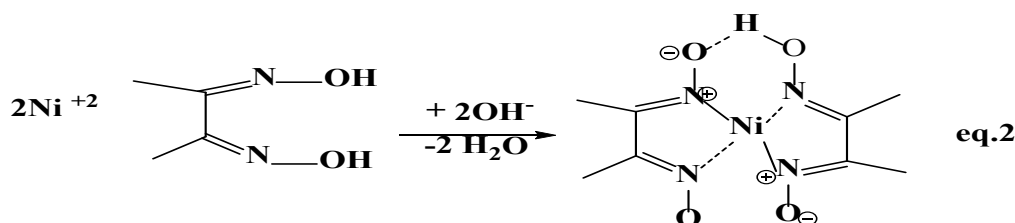as the optimum and economic digestion time for leaching the studied metals from ash using 0.3 M citric acid.

Metals Separation Procedures

For vanadium, nickel and uranium separations, a stock solution was prepared under the optimized digestion conditions (100 g ash sample with 1L citric acid 0.3M at 85°C for 4h). The produced solution contained fundamentally V, Ni, Fe, Na, U, Mg and Zn ions (**Table 2**). The leach liquor was then subjected to successive Ni, V and U separation steps. Due to the high nickel and vanadium percentages in the resultant liquor were subjected to precipitation using complexing agents while uranium content in the leach liquor was only about 48.8 ppm then subjected to a solvent extraction step using 10% TBP/kerosene.

Nickel precipitation

Nickel species in the leach liquor was subjected to a selective separation step using di-methyl glyoxime (*DMG*) reagent (prepared by dissolving 1.16g DMG in 100 ml ethanol). The obtained citrate solution pH was adjusted at 6-7 using ammonia solution before DMG as precipitating agent addition then warmed 50°C for 45 min till complete nickel precipitation as $Ni.(DMG)_2$ (*red precipitate*). The solution after cooling was filtrated then the precipitate was gently washed with double distilled water and dried at 100 °C for 60 min as shown in the flow-sheet **Figure 8**.

In general, nickel in the aqueous solution is present as divalent (Ni^{+2}) which can form a selective complex with DMG with 1:2 ratio. Moreover, the complexation of Ni^{+2} with DMG involves the loss of a proton, so the rate and extent of complexation are pH-dependent. The chelation reaction based on the electron pairs donation on nitrogen atoms, not by the oxygen electrons as presented in equation 2 **[34]**.

The obtained citrate medium, the optimum pH for Ni species precipitation using DMG was 6-9. Decreasing the medium pH than 5 was avoided due to a back $Ni-(DMG)_2$ complex dissolution into the mother leach liquor. On the other hand, the dissolved iron species in the leach liquor which can interfere with nickel ions not precipitated due to presence of citrate ions.

Uranium extraction

The obtained solution after nickel precipitation contained uranium as U (IV) due to the reducing action of citric acid. Tri-n-butyl phosphate (TBP) is the most frequently extractant used due to its high selectivity for uranium species in the reducing medium in the presence beside its high chemical stability and its low toxicity **[35-36]**. Another significant advantage of TBP using for uranium extraction is the ease uranium stripping using water or dilute acids **[37]**. Tri-n-butyl phosphate-containing P=O bond, has a good ability to form complex with uranium (equation 3).

$$UO_2 + 2TBP \rightarrow UO_2(TBP)2 \qquad eq.3$$
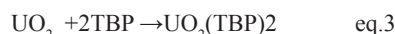
For the uranium extraction studies, 10 ml aqueous solution containing uranium after nickel precipitation was shaken with 10 ml of organic phase containing 10% (v/v) TBP (*Sigma-Aldrich Co.*) diluted with kerosene (*Misr Petroleum Co.*) for 10 min. at room temperature in 50 ml glass separating funnel **[38]**, then the phases were separated and the aqueous phase was subjected to metals analysis. Under these conditions, about 75.2% of the leached uranium species was extracted using TBP/kerosene. After separation, the organic phase was then treated with 10% (v/v) HNO_3 solution as stripping agent for 10 min at A/O phase ratio of 2/1 for stripping uranium. Based on these conditions 85% from the loaded uranium were stripped after 2 stripping stages.

TABLE 2. Metals oxides content in the leach liquor after citric acid leaching.

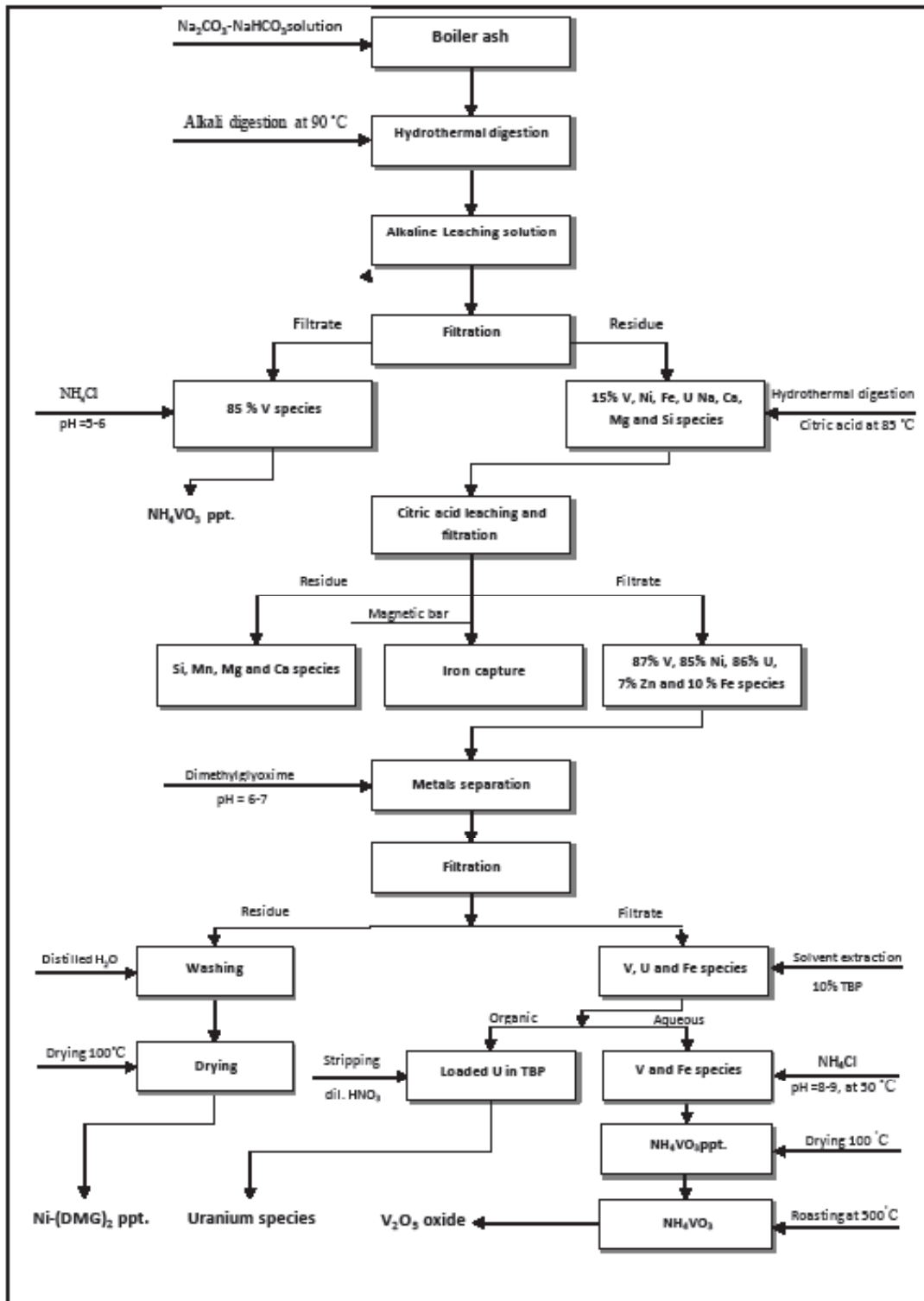| Element oxide	NiO	Al_2O_3	FeO	K_2O	CaO	Na_2O	V_2O_5	ZnO
g/l	11.27	0.78	3.04	1.12	0.96	15.23	2.94	0.91

Fig.9. Flow-sheet of nickel, uranium and vanadium leaching and separation from treated boiler ash.

Vanadium precipitation

After nickel and uranium separations, the remaining liquor solution pH was re-adjusted at 8-9 using dilute ammonia and nitric acid solutions for vanadium precipitation using ammonium chloride (NH_4Cl). The solution was heated under stirring for 20 h at 50°C to precipitate the leached vanadium species as ammonium meta-metavanadate (NH_4VO_3). The resultant precipitate after filtration and washing carefully with distilled water was dried at 100°C for 2h then roasted at 500°C for 2h as presented in **Figure 9**.

Conclusion

In this work, a modified method was applied for dissolution and separation of nickel, vanadium and uranium species from alkali-treated boiler ash residue. The ash was previously subjected to pr-treatment step using $NaCO_3$-$NaHCO_3$ solution for vanadium recovery. This solution leached 85% from the vanadium content while nickel and iron contents were remaining in the residue. The residue after vanadium dissolution was subjected to hydrothermal digestion step using citric acid for Ni, U besides the remaining vanadium dissolutions. Maximum nickel (81%), vanadium (85.2%) and uranium (87.1 %) recoveries were obtained using 0.3M citric acid at 10/1 acid/ash phase ratio at 85°C after 4 h. The resultant leach liquor was subjected to Ni precipitation using dimethylglyoxime at 6-7 pH. Secondly, after Ni precipitation the liquor was treated with10% TBP /kerosene solvent for uranium extraction. The organic phase was treated with nitric acid for stripping the loaded uranium species. Finally, the remaining aqueous solution after Ni and U separations was treated with ammonium chloride as a precipitating reagent at pH; 8-9 to separation the dissolved vanadium content as ammonium meta-metavanadate.

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