



Recovery of vanadium from secondary tailing of iron ore by salt roasting-alkaline leaching and solvent extraction processes

Rahman Ahmadi*, Behnam Sheykhi

Department of Mining Engineering, Imam Khomeini International University (IKIU), Qazvin, Iran

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Abstract

In this paper, the recovery of vanadium from the secondary tail deposits of iron ore by salt roasting-alkaline leaching and solvent extraction has been investigated. To extract the vanadium, after the characterizations studies (XRF, SEM, and EPMA analyzes), preliminary leaching was performed using HCl to reduce lime. Subsequently, the solid that was gained from this stage was put in the oven for palletizing and salt roasting (sodium carbonate) at the temperature of 850°C. This sample was leached in the presence of sodium bicarbonate (NaHCO₃), under certain conditions. Solvent extraction experiments were carried out on the solution of carbonated leaching under different conditions. According to SEM studies, the dominant are calcium, vanadium, and phosphorus in the sample. The percentage of Vanadium (V), according to EPMA results in the 20 selected points, was determined as 0.65%. XRF analysis showed that V₂O₅ and CaO contents of sample were 2.04% and 51.72%, respectively. Lime was reduced to 32.59% after initial leaching with HCl. The carbonate leaching resulted in a total recovery of 93.46% at 85 °C for 60 minutes and with 40 g/l of sodium bicarbonate. In addition, under the optimum conditions of pH=6, the ratio $V_{aq}/V_{org}=1$ and the organic phase concentration of 0.5 M using the anionic Aliquat 336 extractant, Vanadium can be recovered effectively (extraction efficiency=97%) from alkaline leaching solution in the form of decavanadate $V_4O_{12}^{4-}$ or $V_{10}O_{28}^{6-}$ and the anions in the form of VO_3^- or $V_3O_9^{3-}$ in one stage.

Keywords: Vanadium, Alkaline leaching, Solvent extraction, Effective parameter.

1. Introduction

Vanadium is present in over 50 different minerals and is the 22nd most abundant element in the Earth's crust. Vanadinite [Cl(VO₄)₃]Pb₅, Patronite, Carnotyte and Titanomagnetite can be named as the most important minerals in vanadium, also. Vanadium is a metal with high melting point and it has high corrosion resistance against acids at low temperatures and is used as one of the most important alloys in the steel industry (Cheraghi and Shaker-Ardakani 2015). About 88% of vanadium worldwide is extracted from vanadium-containing titaniferous magnetites while the rest is extracted from other minerals such as stone coal or secondary resources such as spent catalyst (Yilong et al. 2017). Generally, Vanadium extraction methods are divided into two groups of pyro-hydrometallurgy and hydrometallurgy from its extractable sources such as Secondary tailing of iron (67% of production, Fig. 1), Vanadium containing ores (21% of production), nuclear fuels and used catalysts (12% Production) (Xiang-yang et al. 2010). In pyro-hydrometallurgical processes, a pyrometallurgical step is initially called as alkali melting or salt-roasting to heat the sample to provide the conditions of extraction. Then hydrometallurgical operations are carried out. The salt roasting of vanadium oxides at a temperature of 800 to 1100 °C leads to the production of met vanadate

phases, such as NaVO₃ sodium metavanadate, which have more solubility (Tavakolikhaleidi 2014). Alkaline leaching is preferred in hydrometallurgical operations by reducing the impurities in the leaching solution, reducing the equipment corrosion, direct discharge of wastewater and solid waste after a simple step of treating acidic leaching (He et al. 2007). The leaching solutions are obtained from hydro-metallurgical and pyro-hydrometallurgical methods have impurities such as silica, aluminum, calcite, iron, and Phosphorus. In addition, these solutions do not have sufficient concentration of vanadium (V₂O₅>15 g/l) (Ye 2006). Therefore, to remove the mentioned impurities and increase the concentration, different purification methods such as settlement (Vitolo et al. 2000), ionic exchange (Tokuyama et al. 2003) and solvent extraction (Tokuyama et al. 2003) are used. Vanadium (V) can form the anion species in the mildly acidic and basic range of pH; therefore, anion extractant solvents are popular in this range. Much research has been done using anion extractants for vanadium solvent extraction (Meawad et al. 2010). Olazabal et al. (1992) reported vanadium extraction using various amine extractants dissolved in Toluene. Vanadium (V) can be extracted by Alamine 336/Toluene in a wide pH range where mono and polynuclear species of vanadium are present. In addition, Aliquat 336/Toluene quantitatively extracted vanadium in the range 3.5<pH<9. The extraction with quaternary ammonium salt is favored in the region

*Corresponding author.

E-mail address (es): r.ahmadi32@gmail.com

where aqueous polynuclear species are predominant. In addition, Necvor and Scheretrova (Nekovár et al. 2000) found that vanadium (V) at low pH (in the range of approximately 2 to 6) was polymerized to the $V_{10}O_{28-n}(OH)^{(6-n)-}$ ($n=0, 1, 2$) ions. The Primene JMT can extract vanadium in this range efficiently. Bale et al. (Bal et al. 2004) showed that vanadium (V) in the acidic range of decavanadate species is rapidly extracted by an anion exchange mechanism with Aliquat 336 in kerosene modified by n-decanol. In another research, El-Nadi et al. (2009) studied the solvent extraction of vanadium (V) of spent catalysts from hydrochloric acid and sodium hydroxide media was separately carried out using an Aliquat-336 extractant dissolved in kerosene containing 10% n-octanol as a phase modifier. The results of these studies showed that the extraction of sodium hydroxide solution is better than a hydrochloric acid solution. Sissy long and colleagues (Sisi et al. 2014) developed a two-stage purification – extraction – stripping - NH_4NO_3 technology to precipitate V_2O_5 based on the characteristics of the alkaline leaching solution produced from roasted stone coal. The purity of the V_2O_5 product was 99.6%, there by meeting national standards. The overall vanadium recovery was 90.9%. In some studies, vanadium was extracted from solutions containing another metal such as chromium, nickel, and molybdenum by Aliquat 336 in kerosene (Nayl and Aly 2015). Also, the extraction of vanadium and molybdenum from liquor solutions was studied by the solvent extraction method. Vanadium and molybdenum can be present in different species depending on the pH of the solution, the concentration and oxide state of the metal and the organic phase (Zeng and Yong-Cheng 2009).

In the present paper, an attempt has been made for the recovery of vanadium using salt roasting - alkaline leaching and solvent extraction methods. The conditions have been optimized for the extraction of vanadium from this sample.



Fig 1. Secondary tailing of iron ore after melting

2. Materials, methods and equipment

The sample required for experiments was prepared from the iron ore secondary tailings of Esfahan Steel Company. Secondary tailing is a side product of steel industries which the amount of it is a little more than 15%-20% of produced steel. This sample is taken from Esfahan Steel Company convertor which is has a stockpile about 15 million tons as depot. The annual produce amount of this Secondary tailing is between 700 to 800 thousand tons. For sampling from the depot, all samples has been taken from different parts of it. After crushing and homogenization, all samples have been divided and a representative sample has been chosen to do different tests. HCl with the purity of 37% in primary leaching, Sodium carbonate (Na_2CO_3) in a palletizing process and sodium bicarbonate ($NaHCO_3$) were used in the alkaline leaching process and sulfuric acid with 98% purity was applied to adjust the pH. Amberlite LA-2, Alamine 336 and Aliquat 336 as extractants, and kerosene with 95% purity, and decanol-1 to 99% purity were used in the solvent extraction process. All the chemical materials were manufactured by Merck of Germany the main sample of slag was grind with a ball mill to $d_{80}=75 \mu m$ and a representative sample of it was analyzed by XRF, SEM, and EPMA analysis. In order to reduce the high amount of lime, 1 kg of a ground sample was first leached with hydrochloric acid in pH=9.5 for 3 hours. the residual soide after mixing with determined values of sodium carbonate (ratio of 0.5) and pelletizing, was roasted in an electric muffle furnace at a temperature of $850^\circ C$ for 60 minutes. Pelletizing was carried out on a rotating disc. The size of the pellets was usually 10 mm. The roasted pellets then cooled and was ground to $d_{80}=75 \mu m$.

After crushing the sample of slag in ball mill, the crushed Secondary tailing with sodium carbonate are mixed and homogenized for 10 minutes in a ceramic mill which includes 20 alumina balls in the diameter of 20mm and 10 big alumina balls which have the diameter of 29mm. A pelletizing machine has been used which has a discus with 1mm inner diameter and is installed in an angle of 45 to horizon, the discus orbits 18rpm to produce pellet (nodal). By adding enough water, the pellets in size of 10mm has been produced because of circular movement of discus. Alkaline leaching experiments were conducted with different amounts of sodium bicarbonate, temperature and leaching time. The stirring rate and solid-liquid ratio were fixed at 280 rpm and 0.05 respectively. Vanadium solvent extraction tests were performed by using different anionic amine extractants in different phase ratios, pH values, and various concentrations. Kerosene was used as a diluent of organic phases with 1-decanol with a specific volume ratio. A mechanical mixer was used to mix organic and aqueous phases. The aqueous phase was analyzed after separation from the organic phase by separator funnel and the recovery was calculated.

3. Results and discussions

3.1. Chemical analysis

The chemical analysis of sample is presented in Table 1. According to the results of the analysis, the amount of vanadium (V_2O_5) in the slag was 2.04%. The amount of lime in the sample is also high and includes more than 50% of the sample. In addition, the amount of silica is also significant.

3.2. Scanning Electron Microscope (SEM)

In order to identify the different elements and the relationship that they have with each other, the points were named EDS (1-5) were considered in the image. In the bright points designated EDS1, there is pure iron with a degree of purity of 86%. In the EDS2 region, which is seen in bright gray, elements such as iron, titanium, manganese, phosphorus, calcium, and vanadium are next to each other, and the elements of calcium, vanadium, and phosphorus are most frequent. In the EDS3 region where looks darker, the elements of silicon, phosphorus, and calcium are contiguous to each other. In this area, the frequency of calcium is higher than silicon and the frequency of silicon is higher than phosphorus. In the area of EDS4, calcium and silicon are adjacent to each other and calcium is more silicon. In the EDS5 region, calcium, iron, and manganese are the main elements, respectively (Fig. 2).

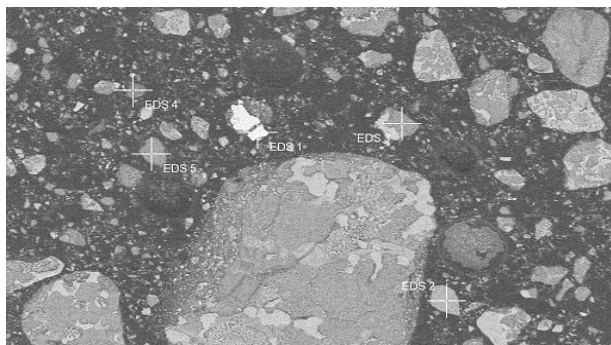


Fig 2. Selected Areas to analyze the existing elements

3.3. Electron Probe Micro-Analyzer (EPMA)

In the EPMA analysis, 20 different points were analyzed. The results show varying amounts of aluminum, silicon, calcium, titanium, manganese, iron, phosphorus, vanadium, and sulfur in all regions. The highest amount of vanadium (V_2O_5) is in the region of 11 and near 3.08%, and then regions 7, 8 and 12 contain 2.58, 2.55 and 2.17% of vanadium respectively (Fig. 3). On the basis of the semi-quantitative analysis, the mean value of vanadium (V) was measured as 0.65% ($V_2O_5 = 2.32\%$) at the 20 points.

3.4. Preliminary leaching

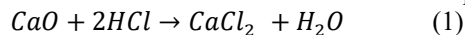
Based on the results of chemical analysis, the amount of vanadium (V_2O_5) and lime (CaO) were measured to be 2.04% and 51.72% respectively. The percentage of V_2O_5 and CaO compounds in the solid after initial leaching was determined 2.42% and 32.59% respectively. A significant portion of lime was removed during the acidic leaching process with hydrochloric acid, and the amount of lime was reduced from 51.72% to 32.59%. The final solid weight decreased from 1 kg to 830gr. Regarding the weight of solid and a percent of V_2O_5 , the recovery of vanadium in the primary leaching stage (acid washing) was 98.46%.

The recovery of primary leaching stage

$$R_{HCl} = \frac{830}{1000} \times \frac{2.42}{2.04} \times 100 = 98.46\%$$

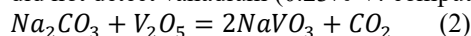
830 is the weight of solid after leaching, 1000 is the weight of primitive sample, 2.42 is the grade of V_2O_5 after leaching, 2.04 is the grade of V_2O_5 in feed and primitive sample.

Free lime in basic materials of vanadium during formation of Vanadium helps to form insoluble vanadates such as calcium meta-vanadate $Ca(VO_3)_2$, calcium pyro vanadate $Ca_2V_2O_7$ and calcium orthovanadate $Ca_3(VO_4)_2$, instead of sodium vanadate soluble in the water (Ye 2006). With the preliminary leaching operations, calcium removal can be carried out according to Equation 1. The $CaCl_2$ is soluble in water, and thus calcium is removed from the sample.



3.5. Alkaline Leaching

By roasting (Fig. 4), according to the equation 2 vanadium is converted to sodium vanadate in aqueous solution. The pH of the leached solution at this stage was about 10 (9.98). In the filtration cake, XRF analysis did not detect vanadium (0.23% V: computational).



In the leaching solution sample, which was determined by ICP analysis, the amount of vanadium (V) was 283ppm (1010.7 mg/l V_2O_5). The vanadium recovery of alkaline leaching was obtained equal to 94.93%. Including two recoveries, preliminary leaching and alkaline leaching in the presence of sodium bicarbonate, overall recovery of roasting/leaching was achieved at 93.46%, which is very desirable.

$$R = \frac{14.6 \times 1010.71 \div 1000}{830 \times 0.7739 \times 2.42 \div 100} \times 100 = 94.93\%$$

3.6. Effect of leachant concentration

Figure 5 shows the effect of sodium bicarbonate concentration on the recovery of vanadium. According to the figure, with the increase of sodium bicarbonate

Table 1. XRF results of chemical composition of the sample

Constituent	MgO	TiO ₂	P ₂ O ₅	V ₂ O ₅	SiO ₂	Al ₂ O ₃	MnO	Fe ₂ O ₃	CaO
Wt (%)	1.67	2.48	2.31	2.04	13.06	1.53	6.33	18.33	51.72

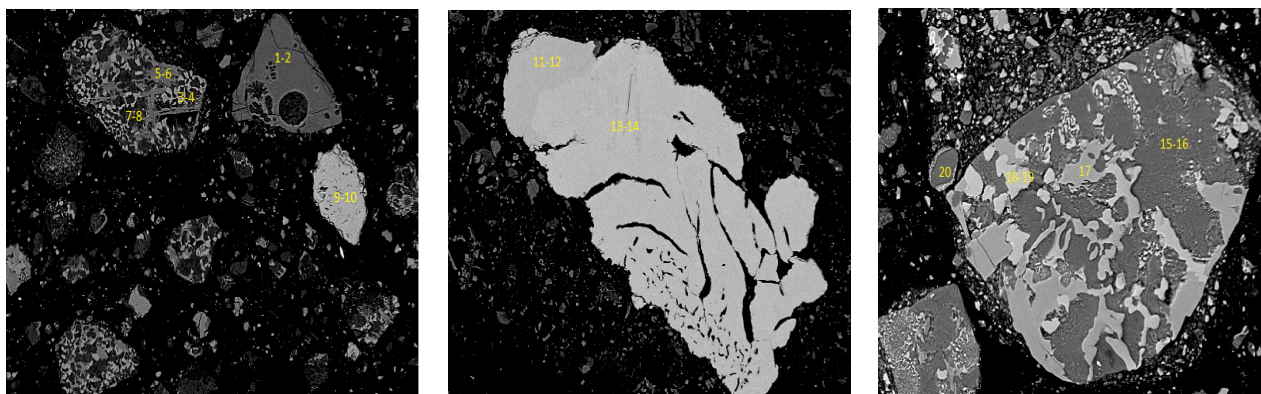


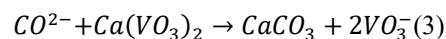
Fig 3. Electron Microscope (BSE) images of the minerals in the example



Fig 4. The size distribution of the pellets, before (right) and after (left) of the roasting process

concentration up to 40 g/l, the recovery of vanadium has reached about 95%. A further increase in the concentration from 40 to 90 g/l did not show any significant change in the recovery of vanadium. Therefore, the concentration of 40 g/l of sodium bicarbonate was selected as the optimum concentration. Carbonate solutions as leaching factors play a very important role in the dissolution of most of the calcium vanadates in the roasting slag. This factor increases the

recovery of vanadium significantly. In this case, the existing calcium vanadium reacts with carbonate ions in the solution that is dissolved by the decomposition of sodium carbonate and formed into the calcium carbonate during the leaching process. The proposed reactions for carbonate leaching are as follows Yosefi (2008):



3.7. Effect of leaching temperature and time

Figure 6 shows the effect of the temperature of the leaching process in different time. According to the figure, increasing the temperature from 50°C to 85°C has increased the recovery of vanadium. Therefore, it can be said that the reaction of vanadium dissolution is an endothermic reaction. In addition, at all temperatures, with an increase in leaching time up to 60 minutes, the recovery increased and then, with increasing of the dissolution time, the recovery was almost constant. Thus, according to the results of Fig. 6, the optimum leaching time and temperature were selected as 60 minutes and 85 °C respectively.

4. Solvent extraction using anion extractants

4.1. Effect of solvent extractant type

Figure 7 shows the extraction of vanadium by three types of anion extractants in different pH values. Aliquat 336 showed increasing vanadium recovery efficiency with increasing pH while Alamine 336 and Amberlite LA-2 showed a sharp optimum at pH=3.5 and pH=2.5 respectively.

According to figure 7, by increasing pH, the extraction rate of vanadium was increased by Aliquat 336 and at pH = 6, the maximum recovery was 85%. These results are consistent with earlier studies for Alamine (Olazabal et al. 1992; Lozano and Godínez 2003) and Amberlite (Rao and Khopkar 1992). Additionally, the extraction rate by Amberlite-LA2 is higher than Alamine 336.

Amberlite-LA2 in the range of pH=1.5-3 and Alamine 336, in the range of pH=3-4 have been extracted vanadium better than the Aliquat 336. The superiority of Alamine 336 on Aliquat 336 in acidic pH was also confirmed by Mewad in 2010 (Meawad et al. 2010). Extraction of vanadium according to table 2 depends on the presence of different species of an anionic and cationic solution at the different pHs.

Aliquat 336, which is the quaternary ammonium salt, is capable to extract different types of anionic vanadium according to reactions 4 to 8 in an extended range of pH (acid to medium alkali) (Sadanandam et al. 1996)

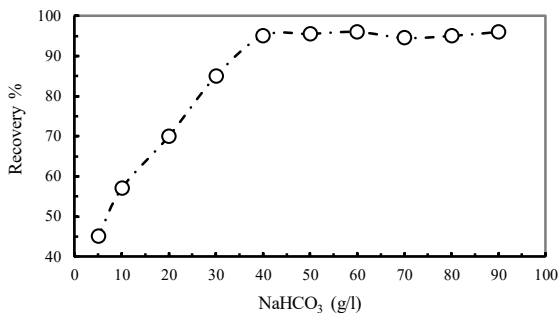
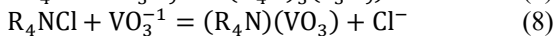
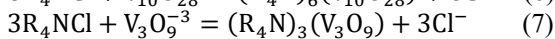
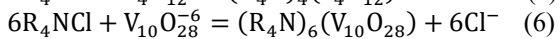
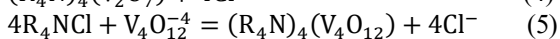
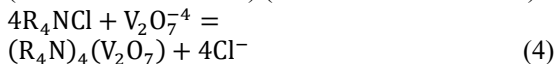


Fig 5. The effect of concentration of sodium bicarbonate on vanadium recovery (T: 85 °C, solid to liquid ratio: 0.05, stirring rate: 280 rpm, d_{80} : 75 μ m)

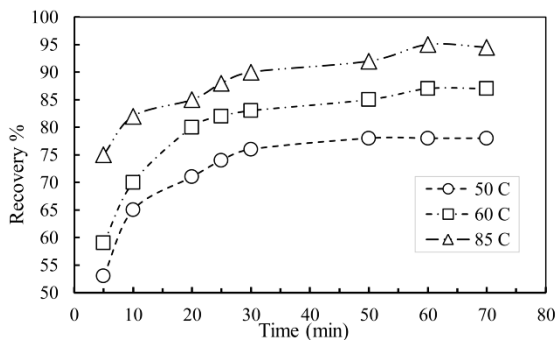


Fig 6. The effect of leaching temperature on the recovery rate of vanadium at different times (Sodium bicarbonate: 40 g/l, solid to liquid ratio: 0.05, stirring rate: 280 rpm, d_{80} : 75 μ m)

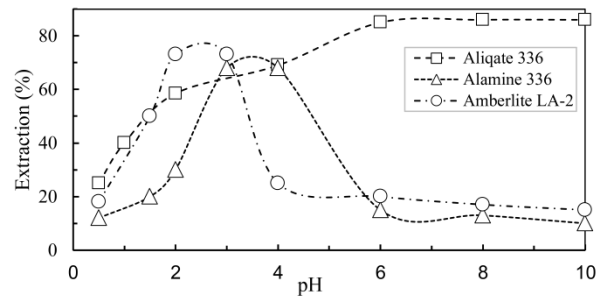
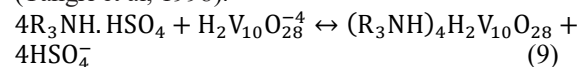


Fig 7. The effect of the extractant type on vanadium extraction at different pHs ($V_{aq}/V_{org} = 1$, extractant concentration: 0.1 M)

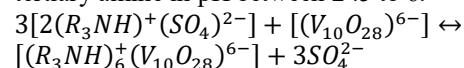
Table 2. Characteristics of some vanadium species in aqueous solutions (Zeng and Yong-Cheng 2009).

Oxidative state	Species	Medium	colour
V (II)	$[V(H_2O)_6]^{+2}$	Acidic	Purple
V (III)	$[V(H_2O)_6]^{+2}$	Acidic	Green
V (IV)	VO^{+2}	Acidic	Blue
V (V)	VO_4^{-4}	Acidic	
	VO_4^{-3}	PH>12.6	A chromaticity
	$V_2O_7^{-4}$	PH=9.6-10	A chromaticity
	VO_3^{-}	PH=7-7.5	
	$V_3O_9^{-3}$	PH=7-7.5	A chromaticity
	$V_4O_{12}^{-4}$	PH=7-7.5	
	$V_{10}O_{28}^{-6}$	PH=2-6.5	Orange-Red
	VO_2^{+}	PH=1-2	Yellow

Regarding the maximum extraction of Amberlite and Alamine 336 at pHs ≤ 4 , it seems that according to Table 2, these extractants are mainly able to extract vanadium only as $V_{10}O_{28}^{6-}$ or $H_2V_{10}O_{28}^{4-}$ under the mentioned conditions in accordance with reactions 8 and 9. Trangeri et al. Described vanadium extraction reactions by Alamine 336 due to the following equation (Tangri et al, 1998):



Lozano and Juan (2001) have provided the following equation as an equation to extract Vanadium (V) by the tertiary amine in pH between 2.5 to 6.



Since the roasted slag leaching, by using alkaline reagents and solvents (sodium carbonate in roasting and in sodium bicarbonate in leaching) has a high selectivity for vanadium dissolution and is suitable for recovery, therefore, Aliquat 336, which is amine of type IV has a good extraction efficiency in the medium alkaline and acid range. In order to extract the vanadium is appropriate as decavanadate $V_{10}O_{28}^{6-}$ or $V_4O_{12}^{4-}$ and anions of VO_3^{-} and $V_3O_9^{3-}$. The results of previous studies have shown the extraction of vanadium anionic polynuclear species at pH values between 2 and 9 by Aliquat 336 (Olazabal et al. 1992; Bal et al. 2004; Zeng and Yong-Cheng 2009).

4.2. Effect of phase ratio: V_{aq}/V_{org}

Figure 8 shows the effect of the phase ratio (aqua-to-organic ratio) of three types of extractants on the recovery of vanadium from the leaching solution. According to the figure, by increasing the ratio of the aqua phase to the organic phase, the percentage of extraction of vanadium was reduced by all three anionic extractants. Increasing the aqua-to-organic ratio for two extractants La-2 Amberlite and Alamine 336 causes a faster reduction in extraction efficiency than Aliquat 336. By increasing this ratio to 5, the extraction efficiency of the three types of Aliquat 336, Amberlite and Alamine 336 has decreased by 60%, 45%, and 25%, respectively. Thus, due to the maximum percentage of vanadium extraction using Aliquat 336 in the $V_{aq}/V_{org}=1$ phase ratio, this ratio was selected as the optimum value.

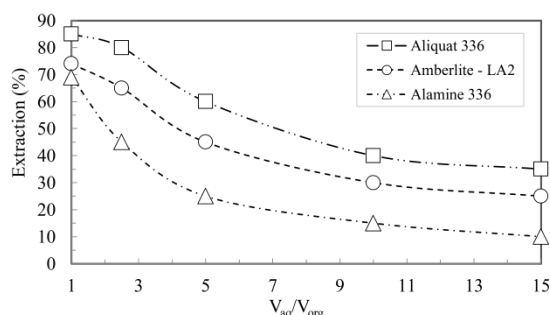


Fig 8. Effect of phase ratio on vanadium extraction efficiency by different extractants (pH=6 for Aliquat 336, pH=3.5 for Alamine 336 and pH = 2.5 for LA-2 Amberlite, Extractants concentration: 0.1 M)

4.3. Effect of organic phase concentration

Figure 9 shows the effect of different concentrations of Aliquat 336 extractant on vanadium extraction efficiency at different pHs and in the aqua-to-organic phase ratio of 1. As shown in Fig. 9, by increasing the organic phase concentration from 0.002 M to 0.5 M, the extraction of vanadium has been increased by Aliquat 336. El-Nadi et al. (2009) have also confirmed the increase in the extraction percent by increasing the concentration of the Aliquat336. According to the figure, at higher pHs, the extraction percentage has reached almost a constant maximum value. Increasing the organic phase concentration from a higher level which causes to increase the viscosity of the organic phase, makes it difficult to separate the organic phase from the aqua phase. Therefore, with respect to the obtained results, by using Aliquat 336 extractant could be a possibility to achieve an extraction efficiency of 97% of alkaline leaching solution in the optimal conditions in which pH= 6, the aqua-to-organic phase ratio:1 and the organic phase concentration = 0.5 M.

4.4. Number of extraction processes - McCabe – Teil diagram

In order to prepare the McCabe-Teil Diagram to predict the number of extraction steps, the extraction isotherm is initially plotted with various proportions of aqua to the organic phase, as shown in Fig.10, and further, the operating line is also plotted according to the concentration of vanadium in the final aqueous solution (Raffinate) which was equal to 30 mg per liter and vanadium concentration in the fresh organic phase was zero and the ratio of aqua to organic ratio was $V_{aq}/V_{org}=1$. The concentration of vanadium in the initial aqua phase was 1000 mg/l. From this point, draw the vertical line from the horizontal axis to cut off the operational line. It was drawn from the horizontal intersection point in the direction in which the isotherm was cut off. Then, from the intersection point, the vertical line that cuts off the operational line was plotted. This process continues until it reaches the end of the operational line. It was found that two theoretical extraction stages are needed to extract almost all the vanadium (V) (Fig. 10) at an V_{aq}/V_{org} of 1. However, with a single extraction stage under these conditions, about 97% of vanadium is extracted, which indicates the proper extraction efficiency.

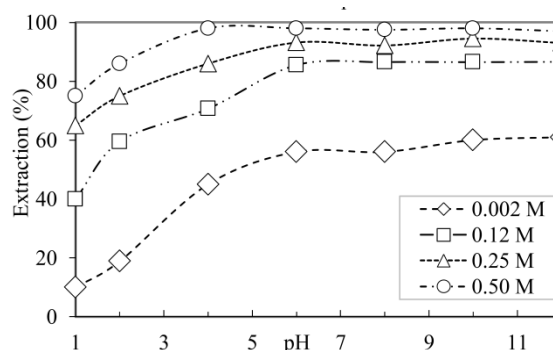


Fig 9. Effect of Concentration of Aliquat 336 extractant on the Efficiency of vanadium extraction at different pHs (aqua to organic phase ratio: 1)

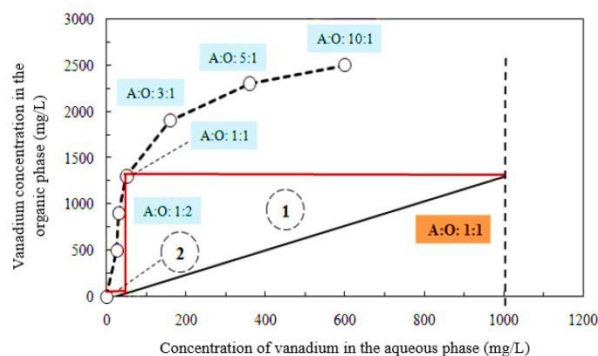


Fig 10. Vanadium (V) extraction distribution isotherm with 0.5 M Aliquat 336 in and its McCabe -Thiele diagram ($V_{aq}/V_{org}=1$)

5. Conclusion

In this paper, the recovery of vanadium from Secondary tailing of iron ore, which is a secondary resource, has been studied by salt roasting - alkaline leaching and carbonated leaching methods. According to the results of the chemical analysis, more than 50% of the slag includes lime. The preliminary leaching results by HCl indicate the reduction for lime (CaO) which was changed from 51.72% to 32.59%. Recovery rate of preliminary leaching was achieved at 46.98%. The carbonate leaching in the presence of sodium bicarbonate (40 gr/lit in 85 °C) has been done on 75 microns-crushed salt in the situation that the pH was about 10 and time was 60 minutes which lead to 94.93% of recovery of vanadium. By increasing pH in tests of solvent extraction, the percent of extraction of vanadium from Aliquot 336 has been raised and reached to maximum and constant rate in the pH=6. However, Alamine 336 and Amberlite LA-2 as two extractor have the maximum rates of extraction in pH=3.5 and pH=2.5, respectively. Furthermore, Amberlite-LA2 as a second type and Alamin336 which is third type Amine have been more efficient than Aliquot 336 between pH=1.5-3 and pH=3-4, respectively. Although in this range, the efficiency of recovery of vanadium by Aliquot 336 is constant. Also, increasing in the concentration of Aliquot from 0.02 to 0.5 M has been increased the percentage of extraction. The results are represented a possibility to obtain 98% efficiency of extraction of vanadium an alkaline leaching solution as decavanadate ($V_{10}O_{28}^{-6}$ or $V_4O_{12}^{-4}$) and the anions which are VO_3^- and $V_3O_9^{3-}$. To reach this efficiency, the pH of solution should be 6, the proportion of aqua phase to organic phase is necessary to be 1 and the concentration of organic phase must be 0.5 molar.

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