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Best Conditions for Extraction and Stripping of Iron from Chloridic Aqueous Medium

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HIGHLIGHTS

- Assessment of extraction technique for separation iron.
- The selectivity of extraction is high compared to other metals.
- With a phase ratio (A/O) (1/1) for the cyclohexane system, the strip solution gave the highest stripping coefficient.

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ABSTRACT

Pure iron is used as an additive for food and drugs to provide the human body with an important element which is iron. For this purpose, iron should be pure and in powder form, the extraction process can supply this type of iron. In this study, extraction of iron from an aqueous solution was done by cyclohexanone as an extractant. The parameters of extraction and stripping processes were studied and assessed the best values which give a high yield of separated iron from ore or any source of iron. For the preliminary study, three general parameters were studied and the best values are agitation time (10 min.), agitation speed (400 rpm), and temperature (30C). In the main study for the extraction process, there are four parameters were investigated and the optimum values are phase ratio (O/A) (1/1), iron concentration (conversely relationship), extractant concentration (proportional relationship), and normality (5 N). The best values for stripping process parameters are phase ratio (A/O) (1/2 – 1/1) and normality of strip solution (0.1 N). The extraction technique is very important in Pharmaceutical industries to produce different suitable metals associated with food and drugs. The extraction technique was detected as an appropriate method to separate iron and introduce it in pure powder form.

1. Introduction

The solvent extraction process is an important process in hydrometallurgy for the separation of metal ions. The extraction process also can be used for many sources of metal such as sea water, industrial waste water and treating of the most waste product like electronic parts. The extraction stage is indicated in Figure1 which shows the whole process for iron production [1].

This technology is now widely adopted for the recovery of metals from low-grade or complex ores [2]. Although the solvent extraction (and leaching) suffers economically from solvent losses, recycling and recovery of side products, the economic challenge for the extraction process may be solved by adding a washing stage to recover small amount of extractants (the cost of extractant higher than acid), as shown in Figure 1 this balancing can solve the economic problem.

The recovery of iron from leach solutions by solvent extraction. The principle of the extraction process is when a metal presents in an aqueous phase is shaken with an organic phase (should have the ability to conjugate with metal ions) metal is extracted, it will be distributed between the two phases. The distribution may be mainly chemical or physical in nature depending on the system (metal and the extractant). Physical processes are those involving the extraction of simple, uncharged covalent molecules. In such cases the extraction (distribution) coefficient, E, is simply the ratio of solute concentration in the two-phase, it is independent of both the total solute (metal) concentration and the phase ratio.

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The stripping process is the reverse process of extraction. Problems can occur when the stability of the extracted complex is so excellent that even concentrated acids will not allow the metal to be stripped. The stability of the extracted species will govern the type and concentration of strip solution required [3].

There are different types of extractants based on structure [4]:

1-Acidic
2- Chelating
3- Solvating
4- Basic

The performance of the extractant is based on the principle of the functional group which is capable to conjugate with metal ions and capture them from the aqueous to the organic phase. Some extractants have more than one functional group this leads to the capability to hold extra ions of metal and gives high capacity for this type of extractants.

Earlier, the extraction process was used for uranium from sea water or source rocks [5] and copper recovery, also can be used to get some trace metal available in some soils [6].

One of the promised applications of metal extraction is the recovery of valuable metals such as gold, silver, platinum and mercury from different sources especially in low concentrations, this application has double benefits: first is the recovery of worth metals, second decreasing the pollution of these elements [7-9]. The advantages of the extraction process for iron can introduce two characteristics, introduce iron in powder form (to be absorbed by the human body) and in pure nature, these two properties are very important in iron using as an additive in food and pharmaceutical industries. Only limited work has been reported on the recovery of iron particularly from low-grade ores such as those found in the area of Al-Husseniat in Iraq.

The extraction of iron is occurred by the leaching of the source of iron (ore or any other sources) by minerals acids (H_2SO_4 or HCl) [10]. The obtained leach liquor is contacted with a suitable extractant in an extraction unit to recover the iron. The extraction unit consist of two stages: the extraction stage and the stripping stage. In this study the extractant is cyclohexanone while the mineral acid is hydrochloric acid. In the stripping stage, the iron will be recovered from organic reagent by acidified water (extremely diluted HCl solution). The value of extraction coefficient depends on many factors: temperature, agitation speed, kinetics of extraction, extractant concentration, phase ratio, acidity, aqueous phase composition, metal ion concentration, organic solvent and dispersion coalescence. To design and operate an extraction unit, the designer should know the parameters how they affect the extraction and the stripping processes to control and select the best values of conditions which give the highest yield of iron.

This study intends to carry out further improvement and optimization to detect the best conditions of the iron extraction process to give high yield for iron powder; the parameters are: (effect of phase ration O/A organic/aqueous, iron concentration, the concentration of extractant and normality of aqueous solution) and for iron stripping process conditions the parameters are: (phase ratio A/O and normality of strip solution).

2. Material and Methodology:

This study is intended to investigate the best conditions of iron recovery in the liquid extraction stage and the stripping stage.

2.1 Chemicals and equipment:

The following chemicals and equipment are used for both stages, extraction and stripping

Chemicals:

1- Cyclohexanone (laboratory cyclohexanone in 2.5 L with purity 99% supplied by ReAgent supplier, U.K)
2- Benzene (laboratory Benzene in 2.5 L with purity 99.5% supplied by McCann Chemicals, U.K)
3- Ferric chloride (laboratory anhydrous ferric chloride in 1 kg bucket with purity 90-99% supplied by Jinan ZZ International Trade Co., Chine)
4- Hydrochloric acid (laboratory hydrochloric acid in 2.5 L with ratio 37% supplied by Sigma-Aldrich Chemie GmbH, Germany)

Equipment's:

1- Water bath equipped with temperature controller, Figure 2 (Digital Heated Laboratory Water Bath with Circulator, model TWBC12-TU3, Australia)
2- Electric mixer with four blade, plastic impeller, Figure 2 (electrical Agitator, model DW-1-60W, Chine)
3- Atomic absorption spectrophotometer, (shimadzu AA-7000, Japan)
4- Glassware (round bottom; 500 ml and separation funnel).

2.2 The experiment procedure

Based on the definition of the extraction (distribution) coefficient, E, is the ratio of solute concentration in the organic phase over solute concentration in the aqueous phase, the removed percent (% Fe) is the solute amount in the organic phase over the amount in origin aqueous solution. The stripping coefficient is the inverse of the extraction coefficient. The parameters of extraction and stripping processes were studied and plotted against these three definitions.

$$\text{Extraction coefficient, E} = \frac{\text{Iron concentration in the organic phase.mol/l}}{\text{Iron concentration in the aqueous phase.mol/l}} \tag{1}$$

$$\text{Stripping coefficient (S)} = \frac{\text{Iron concentration in the aqueous phase.mol/l}}{\text{Iron concentration in the organic phase.mol/l}} \tag{2}$$

$$\text{Fe removed (\%)} = \frac{Ci - \; Cf}{Ci} \times 100\% \tag{3}$$

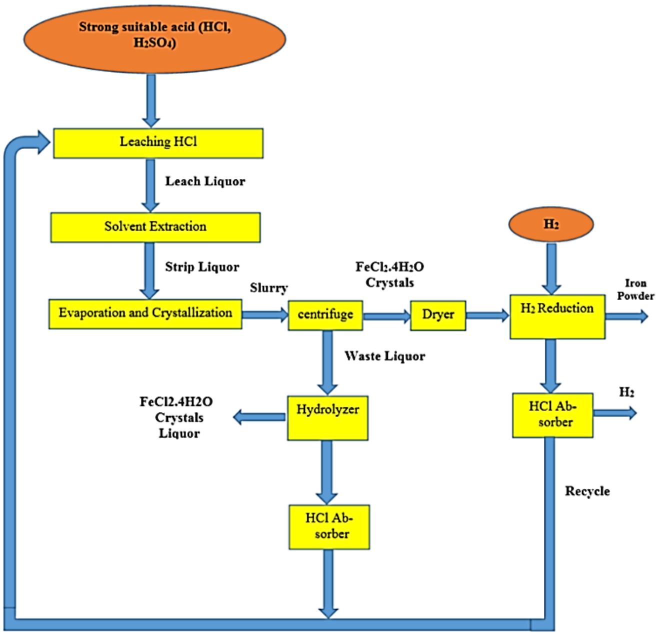

Figure 1: A schematic flow diagram illustrating the practical treatment for iron powder production [1]

Figure 2: Electric mixer with four blades, plastic impeller

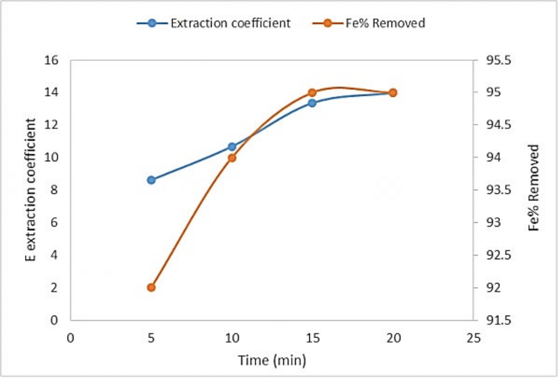

Figure 3: The effect of time vs. extraction coefficient and Fe % removed

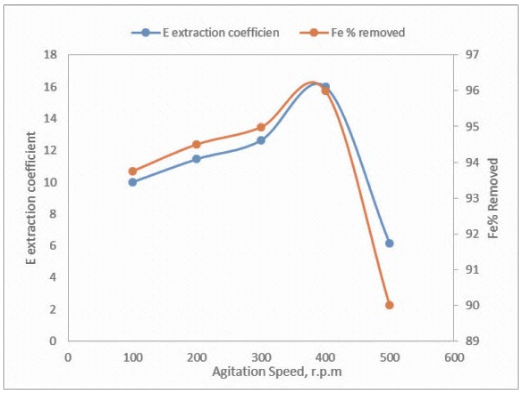

Figure 4: The effect of agitation speed vs. extraction coefficient and Fe % removed

C_i : the initial concentration of iron in the aqueous phase (for extraction process), mol/l
the initial concentration of iron in the organic phase (for stripping process).
C_f : the concentration of iron at the end of the run, mol/l

The aqueous phase was prepared by dissolving $FeCl_3$ in HCI acid solution (4 N) to reach a concentration of iron of about 4 g/L. The organic phase consists of the extractant (cyclohexanone) mixed with benzene as a diluter with a ratio of 50% vol/vol. 20 ml of the aqueous phase was mixed with 60 ml of the organic phase according to the phase ratio equal to 3/1 (as it is fixed in this set). The organic phases were mixed with aqueous phases in a beaker sank in a water bath with a temperature controller. The blades of the mixer enter the beaker to mix the two phases. At the end of each run the content of the beaker was transferred to a separating funnel with a suitable volume and draw the aqueous solution from the bottom and sent to the atomic absorbance spectrophotometer to analyze the concentration of Fe. The preliminary three parameters (agitation time (5 - 20 min.), agitation speed (100 - 500 rpm) and temperature (25 – 40 ^oC)), these parameters were investigated and determined the best value (give high transfer of Iron through two phases) by fixed values of two factors and manipulate the third. The magnitude of the factor which gave a high transfer of iron was considered and fixed in the test of other parameters and so on. The values of the preliminary study that give high separation will be fixed in the main study, it is considered that the preliminary parameters are independent variables on main study parameters so, the effect of them is proportionally on the other parameters. In the main study, to optimize the solvent extraction process of iron recovery; the following parameters were being studied and assessed, phase ratio (O/A) (1/2 - 4/1), the effect of feed concentration (the initial iron concentration in aqueous) (2 – 10 g/l), the extractant concentration (25% - 100% vol/vol, extractant to diluent) and aqueous normality (1 – 5 N). For the stripping process, the studied parameters were phase ration (A/O) (1/2 - 5/1) and normality of strip solution (0 – 3 N). The stripped iron in the stripping solution can be produced as a powder (pure iron) by evaporation of the liquid medium. The parameters of both processes extraction and stripping were supposed as independent parameters and there is no effect for each one to others.

3. Results and Discussion:

3.1 Preliminary Study:

The preliminary study includes the main parameters affected in both processes extraction and stripping; these parameters will be fixed for the main study because their effect is not conjugate with other parameters.

3.1.1 Effect of Agitation Time:

In this set of experiments, the effect of agitation time was investigated. A sample of the aqueous solution was taken at the end of each period (5, 10, 15, and 20 min) for the extraction stage and analyzed. The results are shown in Figure 3, giving the best value of 10 minutes for the cyclohexanone system. The value of 10 min. is the best and more economic gives suitable percentage removed of Fe (94%) as compared with (92% in 5min. and 95% in 15min.) so the extraction coefficient has a high value because there is enough time to mass transfer from the aqueous phase to the organic phase. The extraction coefficient increased by increasing agitation (contact) time until reach to value non change whatever time increased [11]. The transfer of iron from the aqueous phase to the organic occurs by diffusion, the driving force of diffusion is the difference in concentration (ΔC) in both phases. In some metal extraction processes, equilibrium time quit fast (30 min.), and increasing in time lead to a slight increase in distribution coefficient [12,13]. For others, the equilibrium time is different based on metals, Cu, Zn and Cd take about 1hr. while Mn takes 12 hr because the stability constant is low [14]. The suitable time of transfer Fe through phases reflects some determinations of transfer processes such as viscosity of two phases, surface tension between phases and the nature of conjugation of metal with extractant (stability constant) [15].

3.1.2 Effect of Agitation Speed

In this set of experiments, the effect of agitation speed was tested on the extraction process. A sample of the aqueous solution was taken at the end of each run at a different speed, the values are: 100, 200, 300, 400 and 500 rpm, the time of agitation was fixed as 10 min from previous test. The results are shown in Figure 4 explains the best value of extraction coefficient and percentage removed of Fe at agitation speed is 400 rpm. It is obvious that the extraction coefficient increases by increasing the speed of agitation reaching to certain value then decreases and this result is compatible with the result of Farouq [12]. This behavior of increasing of extract coefficient and then decreasing because at high speed the drops of the organic layer suffer from rapture forming small drops [16]. It could be at high speed the most of organic phase forming a vortex layer with low contact area with aqueous phase instead of forming a disperse drops at a slower speed so, the extraction coefficient decreases.

3.1.3 Effect of Temperature

In this set of experiments, the effect of temperature was investigated. A part of the aqueous solution was taken at the end of each run of different temperatures, viz. 25, 30, 35, and 40 °C and examined. The results are shown in Figure 5, giving the highest value for extraction coefficient at 40 ^oC, but there is a small difference between values of extraction coefficients for temperature values (T=40 ^oC, E= 15.7 and T=30 ^oC, E =14.8). Regarding to economic factor (using energy to heat up the solution), 30°C is the appropriate value will be taken in the subsequent runs.

The temperature has two effects: the first, affected on physical properties of two phases (organic and aqueous) such as viscosity, surface tension and kinetics energy of solute (metal), this effect has a slight increase of the extraction coefficient. The second effect depends on the type of extractant and nature of linking of metal ions with it, if the combination of metal ions with extractant is an exothermic process, the effect of temperature will be inverse as in the extraction of copper (II) from chloride media by Cyanex 923 in kerosene [11] and extraction of iron (III) from hydrochloric acid solutions by tributyl phosphate [17], but if it is endothermic, then the action of temperature is proportional. If the combining of extractant with the metal is physical (neither exothermic nor endothermic), the influence of temperature is moderate, as in this study, extraction of iron by cyclohexanone.

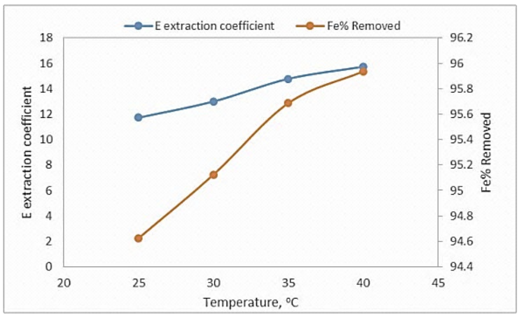

Figure 5: The effect of temperature vs. extraction coefficient and Fe% removed

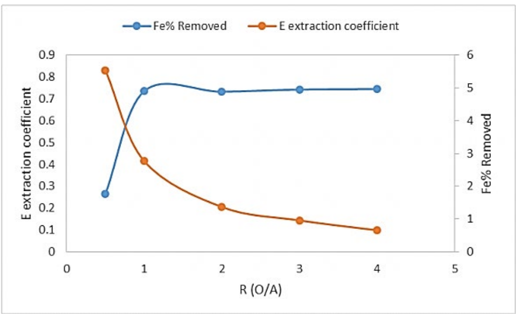

Figure 6: The effect of phase ratio vs. extraction coefficient and Fe % removed

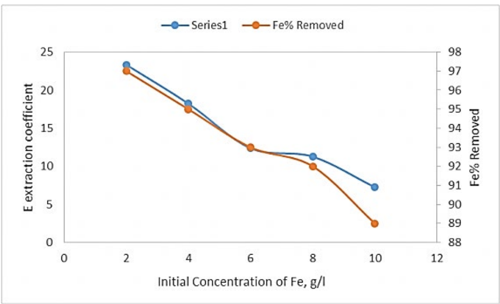

Figure 7: The effect of initial concentration of Fe vs. extraction coefficient and Fe% removed

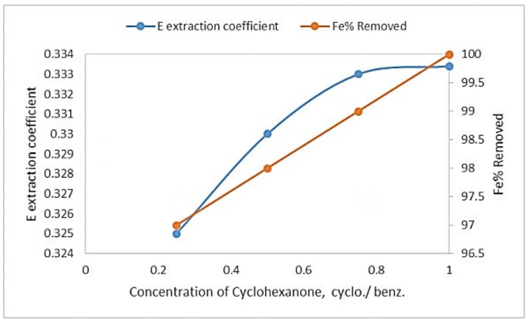

Figure 8: The effect of concentration of cyclohexanone vs. extraction coefficient and Fe % removed

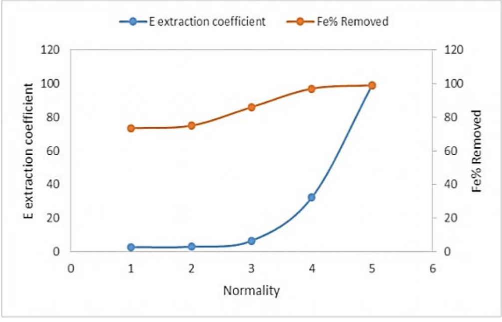

Figure 9: The effect of normality vs. extraction coefficient and Fe % removed

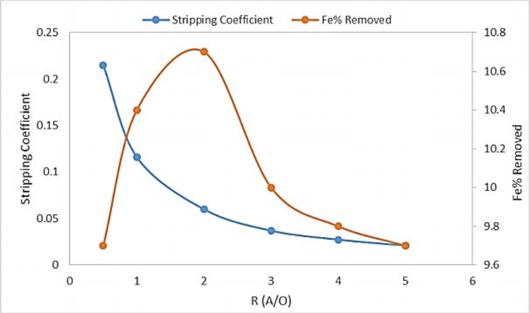

Figure 10: The effect of phase ratio (A/O) vs. stripping coefficient and Fe % removed

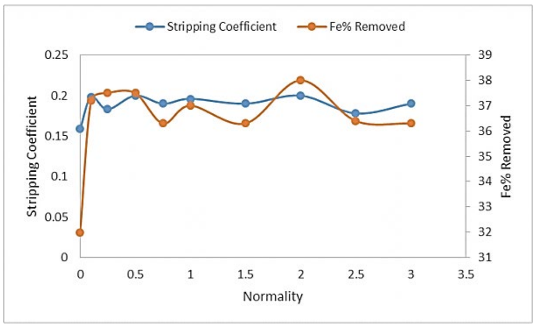

Figure 11: The effect of normality of strip solution on stripping coefficient and Fe % removed

3.2 Main Study

3.2.1 Extraction process

3.2.1.1 Effect of Phase Ratio (O/A):

The obtained results indicate that the increase of the phase ratio (O/A, organic/aqueous layer) causes a significant increase in the extraction coefficient and the phase ratio of 1/1 gives the highest value. This might be attributed to the increase in the quantity of transferred metal which is related to the quantity of extractant that will provide the necessary molecules of extractant to form the complex (the other variables being constants) to reach the equilibrium state. Results were plotted in Figure 6 to further support the above conclusion, where this Figure represents the extraction coefficient and percentage of iron removed vs. phase ratio, the extraction coefficient increases up to a limit then it remains almost constant.

Sokolov et al. [18] studied extraction efficiencies (the same percentage removed) and reach the result that "Extraction efficiencies were almost 100% up to 1:100" the ratio of 1:100 represents extract/aqueous, but this result is not satisfied, a low amount of extractant can capture all solute in a very large amount of aqueous, the right scientific fact is when the ratio be 100:1(a large amount of extractant can hold all metal ions in small amount aqueous solution).

There is an exception for this rule for Mn, the extraction efficiency of Mn decreases by increasing the ratio due to its lower stability constant of complex [14].

The behavior is valid to an extent beyond it the extraction coefficient will decrease, because the quantity or volume of the organic phase increases, the amount of metal transferred will undergo a somewhat smaller increase leading to a decrease in the concentration of metal, hence causing a decrease in the extraction coefficient.

The extraction coefficient (metal concentration) is more important than the quantity of metal because it is significant in the stripping process and for economic consideration for using the minimum affected quantity of extractant.

3.2.1.2 Effect of Iron Concentration

From the result of the previous section as it was noticed, the phase ratio of (1/1) gave the highest extraction coefficient. This phase ratio was used to carry out the experiments in this section. Keeping other variables constant, the results clearly demonstrate that increasing the iron concentration in the feed causes decreasing the extraction coefficient as shown in Figure 7. This conclusion could be drawn from the definition of the extraction coefficient (ratio of solute concentration in the two phases). Knowing that a certain number of extraction molecules are associated with each metal ion, thus upon keeping other variables constants, the metal concentration in the solvent will remain almost constant in spite of its increase in the aqueous phase. This will lead to a reduction in the extraction coefficient. Further support the above conclusion where the percentage of iron removed decreases with the increase of the iron concentration in the feed. The increasing of metal concentration in the feed solution guide to change properties of the continuous phase (aqueous) such as viscosity and density and these effect on dispersing of the organic layer into aqueous layer then control the transfer of metal from aqueous to organic phase [16]. There is another result, which is by increasing the initial concentration of metal, the removed percentage will be increased [12]. The difference of effect for initial metal concentration on percentage removed depends on the type of extractant, some extractants (with multi-function groups) have high capacity (amount of solute certain extract able to collect) however, increasing of metal concentration can be ingested by extractant. Other extractants have a low capacity (have one functional group in charge of grabbing metal ions) so, they reached saturation even with a low concentration of metals, then any increase in concentration will stay in the aqueous solution without transferring to organic.

3.2.1.3 Effect of Extractant Concentration:

The results showed that upon increasing the extractant concentration as the extraction coefficient increased. This could clearly be seen by plotting the extraction coefficient (E) vs. extractant concentration as shown in Figure 8, further support the above conclusion, where the percentage of iron removed plotted vs. extractant concentration. It is obvious that upon increasing the extractant concentration, the percentage of iron removed increased. Devi [11] obtained the same results, at 0.05 M, the extraction of copper was only 23.05 per cent, whereas it rose to 97.5 percent at 1.0 M, in the extraction of iron (III), the percentage increased rapidly from 84.45 to 98.57 % with increasing TBP concentration [13] and for iron extraction by decanol or aliphatic alcohol in ketone the same effect of extractant concentration [15,18]. Heavy metals like Zn, Cd and Mn, their extraction efficiency increased by increasing the 8-HQ concentration [14].

The wide range of values of extraction coefficient obtained using different concentrations (25, 50, 75 and 100 % by dilution with benzene) may be due to the diluent used which could affect the solvation of the extractant and hence, its extractive properties. This result improves the recent conclusion that the Fe molecules conjugated with extractant molecules only without diluted molecules. This phenomenon leads to the fact which is the presence of Fe in the organic phase is not about ordinary diffusion while about conjugate with suitable molecules (extractant molecules). Low numbers of molecules exist in a high volume of the mixture (extractant and dilute) gives a low extractant coefficient and vice versa. The dilution of extractants has some advantages when the viscosity and density of the extractant are high so, it is difficult to get a high mass transfer through a viscous phase for this reason dilution will be an appropriate choice [16].

3.2.1.4 Effect of Normality:

The results obtained here within the range of normality values of the aqueous leach liquor studied (1 - 5 N) both the extraction coefficient and the percentage removed increases with increasing normality (decreasing of pH), this is shown in Figure 9, where the recovery increase with increasing normality values. The result was gotten by Devi [11], in the extraction of copper the removed percentage increases from 6.7 to 78.5% when molarity of HCl increases from 3 to 6.5M and then decreases. The same behavior was investigated in a study of the extraction of iron (III) from HCl solutions by Nouioua [13] and Sato [17] which is the distribution coefficient rises steeply with Tributyl Phosphate as an extractant. Saji [19] refers that the iron III extraction using TBP–MIBK increases with the increase in acid concentration. There is a value of normality of solution gives the maximum extraction coefficient beyond this value of normality the extraction of metal will be decreased. The justification of this behavior of extraction via normality (increasing – level off – decreasing) is due to hydrolysis of the metal increasing of acidity for aqueous solution [20]. At higher acidity (high concentration of H^+) the hydrogen ion will compete with the metal ions and conjugate with extractant instead of ions metal [11].

3.2.2 Stripping Operation:

3.2.2.1 Effect of Phase Ratio (A/O)

The obtained results are shown in Figure 10 indicate that the increasing in the phase ratio (A/O) leads to a decrease in the stripping coefficient. Thus, (1/2 and 1/1) are the best ratio for cyclohexanone. This could be attributed to the increase in the volume of the aqueous phase which will consequently lead to a decrease in the concentration of iron. Knowing that the amounts of iron transferred to the aqueous phase are almost constant as it does not require the complex formation and therefore, it is independent of the amount of aqueous phase used.

3.2.2.2 Effect of Normality of the Strip Solution

The results obtained are plotted in Figure 11, indicate that using an acidic strip solution of 0.1 N of the hydrochloric acid solution gave the highest stripping coefficient (keeping other variables constant). However, increasing the acidity of the strip solution beyond that causing a slightly decrease in the stripping coefficient as noticed from the same Figure. The fluctuation might be attributed to a reverse action due to unstable complex formation between the extracted species and the acid which will lead to a reduction in the stripping coefficient. Similar results were observed by Change [21] in the separation of iron from zirconium in concentrated hydrochloric acid solutions using several types of commercial extractants, also Mao [15] explained the inverse effect of concentration of HCl on stripping coefficient in iron extraction by decanol. The stripping of iron can easily be achieved using dilute HCl as the stripping solution. By evaporation of the solution, pure iron will be produced.

4. Conclusions

The extraction technique is very suitable to separate metals in low concentrations (especially valuable metals). Pure iron powder can be produced in the extraction technique. The best conditions to produce the highest yield of iron powder were studied and the results as below.

For the extraction process a phase ratio (O/A) of 1/1 gave the highest extraction coefficient for the conditions used. The extraction coefficient increased when the concentration of iron decreased in the aqueous feed for given conditions. The extraction coefficient increased when the concentration of extractant increased when other conditions are fixed. Both extraction coefficient and recovery percentage increased with increasing normality, within the range of normality used (1-5 N), the highest value at 5N. In fact, there is a specific value of pH (certain value of Normality) for each metal, the selectivity of extraction is high according to other metals.

For the stripping operation, it was found that using a phase ratio (A/O) about (1/1) for cyclohexanone system and low acidity (0.1 N), the strip solution gave the highest stripping coefficient and percentage of removal.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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