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# Upgrading of Egyptian Oil Shale Using Enhanced Gravity Separation

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**Abstract:** Egyptian oil shale from Wadii El-Nakhil, Red sea region was upgraded using enhanced gravity separation. The oil shale sample was characterized physically and chemically to determine its mineral content and characteristics. The sample includes quartz, siderite, apatite, anhydrite and calcite. The clay mineral is mainly represented by kaolinite while the organic matter is 30%. The ground sample (less than 50 microns) was classified into two fractions. The coarser was higher than 25 µm while the finer was less than 25 µm. The lower and upper levels of both the centrifugal force and water pressure have been suggested to construct the design for Falcon Concentrator type SB-40. The coarse concentrate of 42% kerogen with 94.35% recovery was achieved at 60 Hz (equivalent to G-force 176) and water pressure of 4 Psi from feed of 29% kerogen. The fine concentrate of 38.46% kerogen with 85.4% recovery was achieved at 70 Hz (equivalent to G-force 243) and water pressure of 2 Psi from feed of 33% kerogen.

**Keywords:** Oil Shale, Energy Minerals, Enhanced Gravity Separation, Kerogen, Falcon Concentrator

## 1. Introduction

Oil shale, an organic-rich and fine-grained sedimentary rock, consists of a mineral porous matrix that contains insoluble kerogen. Liquid hydrocarbons (shale oil) and combustible shale gas can be obtained from insoluble kerogen after heat treatment [1-3]. With the severe energy shortage and high energy prices, developing effective and economic methods to utilize oil shale as an alternative energy has been the focus of many countries rich in oil shale [4-6].

The beneficiation of oil shale to recover kerogen enriched products was proposed as early as 1920 by Dolbear [7]. Later, the US Bureau of Mines and others reported work on beneficiation of western oil shale [8-9]. Upgrading of a finely ground oil shale particles in the range of 15-150 µm through flotation and oil agglomeration has been studied [10, 11]. The separation of kerogen rich particles with an efficiency of 70% was obtained. Heavy liquid separation was reported to upgrade oil shale, but the kerogen recovery in the enriched products in most cases ranged from 14% to 50% of the feed [12].

Oil shale may contain 10-35% (by weight) kerogen which decomposes and yields crude oil when heated to 400 - 500°C. The beneficiation step offers the potential of improving the economy of retorting while achieving other benefits such as reducing energy [13-14]. The extremely fine size of the enriched flotation concentrates should favor improved kinetics and chemical reactivity of the shale during the conventional retorting, hydro-retorting or other novel conversion processing. They showed that flotation can reject 50 - 70% (by weight) non-fuel minerals before processing, and improve the productivity of the reactor.

Centrifugal concentrators, also referred to as enhanced gravity concentrators, employ centrifugal force to improve the settling rate of particles. There are at least seven different types of centrifugal gravity separators commercially available and new types are still being developed. The better-known separators are: the Knudsen bowl, Knelson, Falcon SB, Falcon C, Kelsey Jig and the Mozley multi gravity separator. The Knudsen, Knelson, Falcon SB and Falcon C machines are of the same generic type. These machines are best suited for feeds containing a small percentage of high density material. They can be used either for roughing, scavenging or cleaning. Generally, a smaller machine would be used for cleaning on a batch scale. Their main application is for the recovery of gold, but they have potential to recover any mineral that has significantly higher density than the bulk of the feed [15].

Two centrifugal concentrators of Canadian origin, the Knelson Concentrator and the Falcon Concentrator, have gained wide spread application. They are mainly grouped as semi-continuous and continuous types. Conventional gravity separation devices need a minimum relative density differential of at least one between the light and heavy phases for an effective separation whereas for the centrifugal machines, the minimum required density difference is even low. However, the bigger the density differences the better for the separation. These units have been used on pilot scale on different coals in the United States. Falcon Concentrators claim that Falcon C machines are capable of processing 100 t/h solids, recovering particles as fine as 10 µm [16].

In Egypt, Red Sea region the estimated resources of oil shale are approximately 15 billion tons, the discovered reserves of oil shale. The present paper focuses on the enrichment of organic matter of oil shale from Wadii El-Nakhil, Red Sea coast by falcon concentrator as an enhanced gravity separator to obtain concentrates rich in organic matter, which can be used in a retorting process as an energy source.

# 2. Materials and Methods

### 2.1. Oil Shale Sample

A representative sample, 10 Kg ore, of Wadii El-Nakhil sample was subjected to crushing using pilot 5 × 6 "Denver" jaw crusher to 100% −3.3 mm. The crushed product was finely ground to 100% (−50 µm) via “Rod Mill”. The effect of grinding time, number of rods and solid-liquid ratio were studied. Rod mill length = 34 cm, length of rod = 33 cm, diameter of rod = 15 mm, diameter of mill = 14 cm, mass of grinding medium were 2.75 and 5.50 kg for 6 and 12 rods. The mass of feed was about 700 g.

The ground sample was classified into coarser fraction (−50 +25µm) and fine fraction (−25µm).

### 2.2. Characterization of the Oil Shale Sample

The thermal and physical properties of oil shale were studied by a variety of methods such as thermogravimetry (TG), Fourier transform-infrared spectroscopy (FTIR), and X-ray diffraction (XRD). Thermal gravimetric analysis (TG) cannot distinguish the individual thermal properties of either kerogen or mineral. FTIR is usually used to identify the hydrocarbons and indicate the particular group of minerals [17]. XRD patterns can provide comprehensive information on the crystalline structure of the mineral [18-20]. Scanning electron microscopy (SEM) is a type of electron microscope that used to determine the morphology and detailed structural in formation of the samples [21].

Mineralogical analysis of the sample was done using optical microscope on thin and polished sample sections. For the surface and powder characterization, XRD and FTIR studies were performed. Chemical analysis of the oil shale sample was determined using X-ray fluorescence.

### 2.3. Gravity Separation Technique

Falcon SB-40 concentrator is a semi-continuous centrifuge units, generally operating at a relatively higher G-forces, 300G. Falcon SBs are manufactured in different sizes, from laboratory scale to high capacity models. The laboratory unit SB-40, Figure1, has a concentrating surface of roughly 40 square inches, and a diameter of 4 inches. The largest unit, the SB-5200, is capable of treating up to 400 t/h.

The bowl is rotating in a vertical axis. Its lower part is smooth and tapered inwards and it supper part consists of a certain number of fluidized riffles (two for the SB-40) for concentrate collection. The grooves are evenly perforated for back-injection water to fluidize the concentrate bed and they have the same diameter as shown in Figure 2 [22]. The bowl speed can be adjusted so that up to 300 g's of centrifugal force can be produced to cause deposition and stratification of the fine particles against the inside wall of a smooth centrifugal bowl.

Both the coarser fraction (−50 +25 µm) and fine (−25 µm) were used as a feed for Falcon Concentrator type SB-40. The feeding material was introduced as slurry of 10% solids through the central vertical feed pipe with feed flow rate of 0.5 L/min and accelerated by a high speed impeller.

In the retention zone, which was immediately above the migration zone, fluidization water was injected through the rotor wall to create a dilated or fluidized bed. The high specific gravity target particles became embedded in this zone and were retained until the machine was off and the concentrate was rinsed down through the concentrate discharge ports. A centrally located rinse manifold directed jets of water to thoroughly rinse concentrate from the retention zone after each operating cycle. The rinsing process can normally be accomplished in less than 40 seconds. All products were collected, dried, and directed to evaluation.

On the basis of many exploratory tests, the lower and upper levels of both the centrifugal force and water pressure have been suggested to construct the design. Parametric optimization of the separation process has been studied using response surface method (RSM) and central composite rotatable design (CCRD). The design-matrix of different runs, 11 experiments, was illustrated in Tables 1 and 2 [23-25].


***Figure 1.*** *Falcon concentrator model SB-40.*

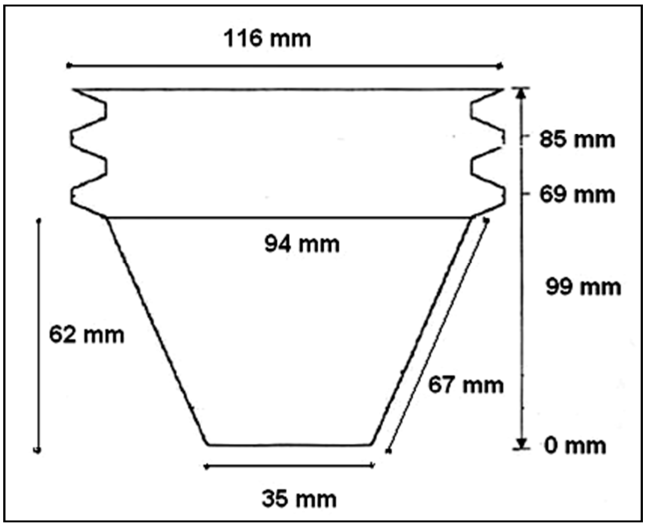


***Figure 2.*** *Sketch of Falcon concentrator model SB-40.*

***Table 1.*** *Box-Behnken design with 3 levels and 2 factor levels of Falcon concentrator tests.*

| Run No. | Coded Factor Levels | |
|---|---|---|
| | A | B |
| 1 | – | – |
| 2 | + | – |
| 3 | + | + |
| 4 | 0 | 0 |
| 5 | 0 | 0 |
| 6 | 0 | + |
| 7 | – | + |
| 8 | 0 | – |
| 9 | + | 0 |
| 10 | – | 0 |
| 11 | – | – |

***Table 2.*** *Factors of Falcon concentrator tests.*

| Factor | Units | Low Actual | High Actual | Low Coded | High Coded |
|---|---|---|---|---|---|
| Frequency | Hz | 60 | 80 | -1.00 | 1.00 |
| Water pressure | Psi | 1.0 | 5.0 | -1.00 | 1.00 |

***Table 3.*** *Falcon data sheet for RPM and G-force.*

| Frequency, Hz | RPM | G-force |
|---|---|---|
| 60 | 1750 | 176 |
| 70 | 2041 | 243 |
| 80 | 2282 | 300 |

# 3. Results and Discussion

## *3.1. Characterization of the Oil Shale Sample*

Mineralogical analysis of the sample showed that the shale matrix is composed of alternating lamina of 10-50 µm thickness. The lamina is consisted mainly of carbonate and argillaceous material that is rich inorganic matter. Quartz occurs as cavity filling of foraminifer's chambers or dispersed within the matrix. Pyrite in oil shale occurs as finely dispersed particles or in framboidal form. Phosphate represented by phosphatic bioclasts including bone and scale remains of vertebrates and fishes, Figure 4. The XRD spectrum of raw Egyptian oil shale confirmed mineralogical analysis [21, 26]. The FTIR spectrum shows that the sample is rich in carbon and oxygen as aliphatic and aromatic matrix. Several distinct and strong aliphatic and aromatic bands confirmed the high organic matter content of the sample, Figure 3. Table 4 shows that the sample has a high content of calcium oxide, sulfur, silica, alumina and iron oxide, Table 4.

***Table 4.*** *Chemical analysis of oil shale sample.*

| Composition | Weight,% |
|---|---|
| $SiO_2$ | 15.34 |
| CaO | 21.05 |
| $Al_2O_3$ | 4.67 |
| $Fe_2O_3$ | 2.31 |
| $P_2O_5$ | 3.08 |
| $SO_3$ | 7.48 |
| L.O.I | 42.9 |

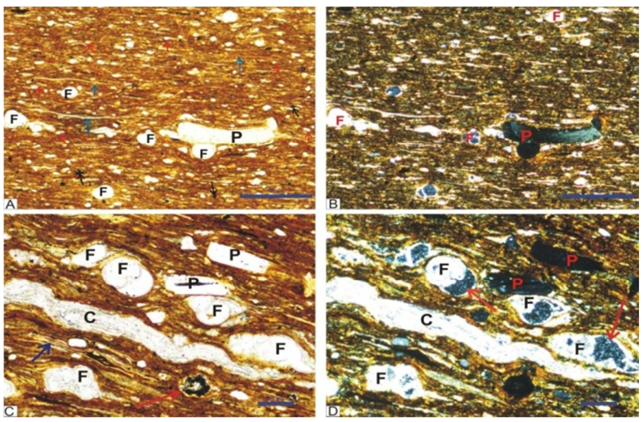

***Figure 3.*** *Thin and polished sections of the oil shale sample.*

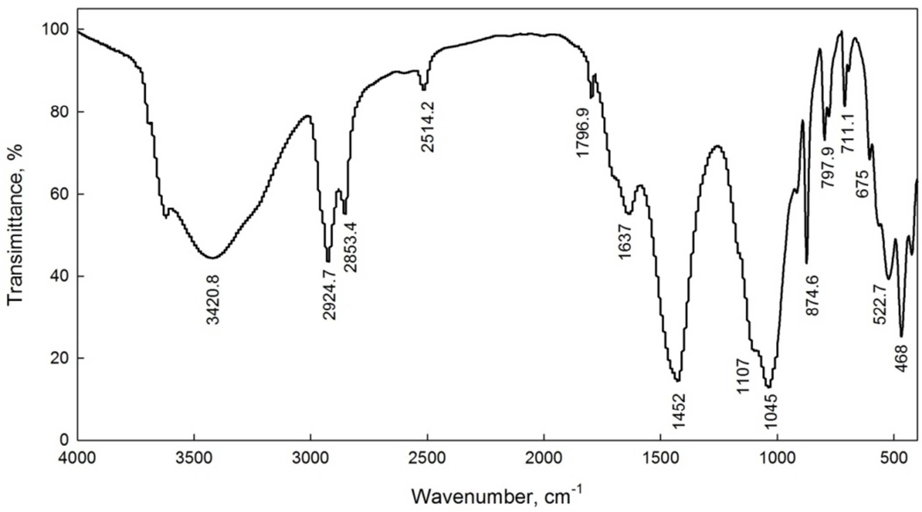


***Figure 4.*** *FTIR spectrum of the Egyptian oil shale sample.*

### *3.2. Grinding Parameters*

Figure 5 shows the effect of grinding time using 6 or 12 rods, the maximum grinding was achieved at 90 min. The weight percent of −50 µm was 26 and 46% for 6 and 12 rods respectively. The grinding was carried out as a wet process to avoid oxidation of kerogen as a result of increasing temperature in dry process. Table 5 shows that the maximum grinding was achieved with solid-liquid ratio of 2:1. The ground product below 50 µm was 30 and 56% for 6 and 12 rods, respectively.

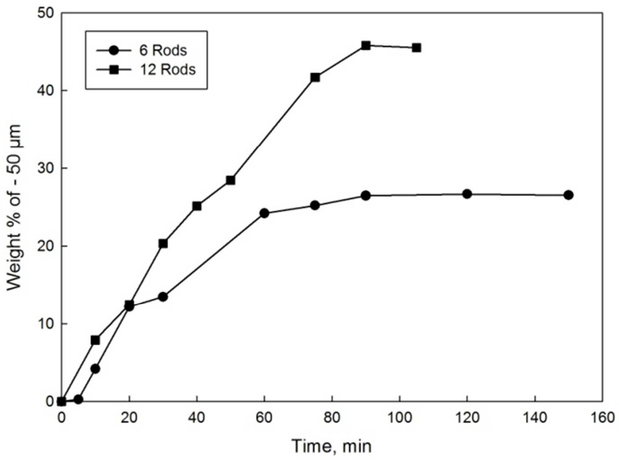


***Figure 5.*** *Effect of time on grinding efficiency of oil shale.*

***Table 5.*** *Effect of solid-liquid ratio on grinding efficiency.*

| S:L ratio | Weight% of –50 µm | |
|---|---|---|
| | 6 rods | 12 rods |
| 1:1 | 26.45 | 45.80 |
| 2:1 | 30.30 | 56.30 |

Table 6 shows that the particle size analysis of ground product with kerogen content. The ground oil shale was classified into a coarser fraction (–50 +25 µm) and a fine (–25 µm).

***Table 6.*** *Size analysis and grade of the ground product.*

| Fractions | Weight% | Kerogen% |
|---|---|---|
| −50 +25 µm | 36.67 | 29.0 |
| −25 µm | 63.33 | 33.0 |
| Head | 100 | 31.5 |

### *3.3. Enhanced Gravity Separation*

Figures 6 – 9 show the contour and 3D graphs for the effect of frequency (G-force) and water pressure of wash water on to kerogen grade. A concentrate of low grade and recovery was obtained at frequency of 60 Hz (176 G-force) and water pressure up to 2.5 psi, while increasing water pressure up to 5 psi increased the grade and the recovery of kerogen. Increasing the G-force up to 243 (70 Hz) increased the grade at moderate water pressure but with moderate recovery. Increasing the G-force up to 300 (80 Hz) increased the grade but with low recovery. The recovery was increased with increasing water pressure up to 5 psi [27].

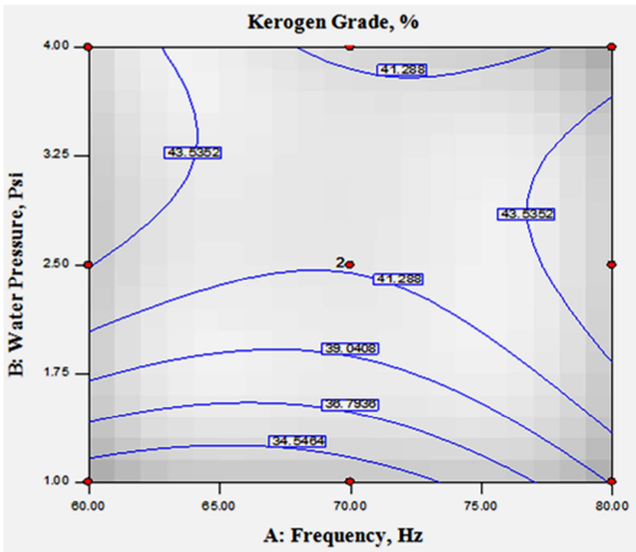


***Figure 6.*** *Effect of frequency and water pressure on Kerogen grade.*

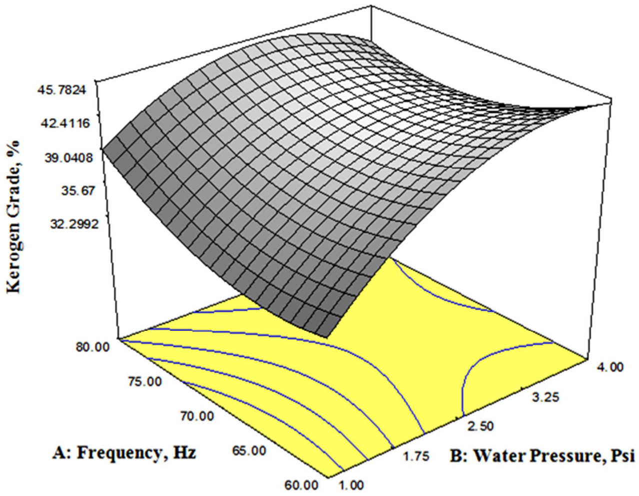


***Figure 7.*** *The 3D of frequency and water pressure on Kerogen grade.*

Table 7 shows the Design layout and experimental results for falcon concentrator test with actual values and predicted results for the coarser fraction (–50 +25 µm). The best result of 43.22% Kerogen with 88.19% recovery was achieved at frequency of 60 Hz and water pressure of 4 Psi. Also, a concentrate of about 42% Kerogen with 92 - 94% recovery was achieved at frequency of 60 - 70 Hz and water pressure of 4 - 5 Psi.

***Table 7.*** *Experimental results for the coarser fraction (–50+25µm).*

| # | Frequency, Hz | Water Pressure, Psi | Kerogen | |
|---|---|---|---|---|
| | | | Grade,% | Recovery,% |
| 1 | 60 | 1.0 | 35.72 | 2.54 |
| 2 | 80 | 1.0 | 42.36 | 34.21 |
| 3 | 80 | 4.0 | 40.92 | 92.14 |
| 4 | 70 | 2.5 | 43.22 | 68.32 |
| 5 | 70 | 2.5 | 43.23 | 68.33 |
| 6 | 70 | 4.0 | 42.44 | 92.45 |
| 7 | 60 | 4.0 | 43.22 | 88.19 |
| 8 | 70 | 1.0 | 26.96 | 3.05 |
| 9 | 80 | 2.5 | 43.11 | 21.32 |
| 10 | 60 | 2.5 | 42.61 | 71.09 |
| 11 | 60 | 5.0 | 42.00 | 94.35 |

The rotation velocities of the Falcon make it able to recover finer dense particles but may hinder coarser one. The Falcon bowl has a limited fluidized groove surface at the top circular portion and a large conical non-fluidized section at the bottom. The lower water pressure was not suitable for escaping the lighter particles (kerogen) from heavier particles which were hold inside the bowel, thus lower recovery was obtained. As a result, the Falcon generally requires more water pressure. In the retention zone, which was immediately above the migration zone, fluidization water creates a dilated or fluidized bed. So, increasing the pressure increased both grade and recovery of kerogen. The maximum grade of 43.2% with recovery of 88.2% was achieved at 60 Hz (equivalent to G-force 176) and 4 Psi.

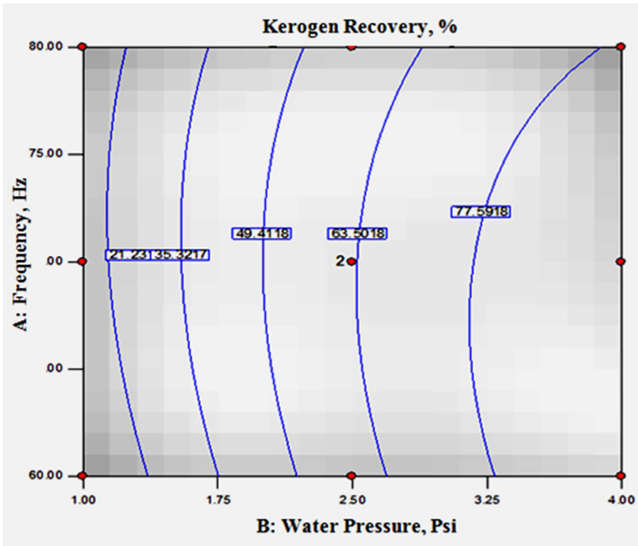


***Figure 8.*** *Effect of frequency and water pressure on recovery.*

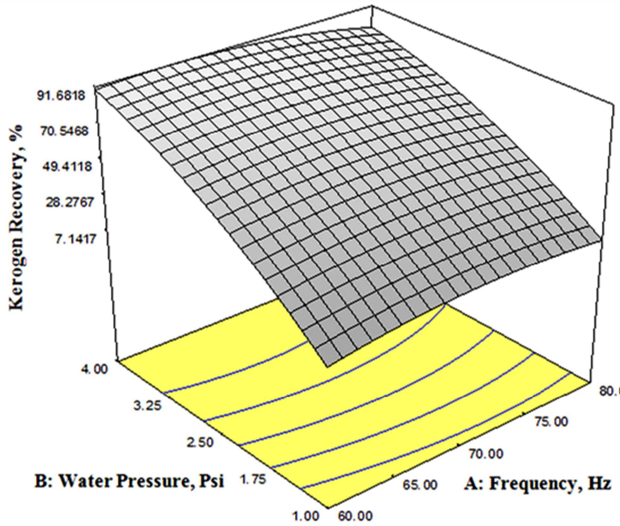


***Figure 9.*** *The 3D of frequency and water pressure on Recovery.*

The correlations between the design factors and the responses were adequately described by polynomial models. Design-Expert 6.0.3 software program was used to analyze each response to the regression model of the factors listed above, using the following methodology: a) analysis of variance (ANOVA) was conducted to determine the adequacy of linear, quadratic and cubic models; b) one model was then chosen for an in-depth regression analysis; c) diagnostic evaluation of the robustness of the model was determined and d) a response surface analysis was conducted to optimize the separation efficiency. The grade and recovery of kerogen can be calculated by using equations (1) and (2) which were derived from the design summary.

$$\text{Kerogen}(\%)= 41.43+(0.75\times A)+(3.85\times B)+(2.91\times A^2)-(4.44\times B^2)-(2.38\times A\times B) \quad (1)$$

$$\text{Recovery}(\%)= 62.91-(1.34\times A)+(37.13\times B)-(5.85\times A^2)-(9.75\times B^2)-(4.38\times A\times B) \quad (2)$$

Where A: Frequency (Hz) and B: Water pressure (Psi)

Table 8 shows the experimental results for the falcon concentrator test with actual values and predicted results for the fine fraction (−25 µm). At frequency of 60 Hz (176 G-force) a low grade was obtained even with higher water pressure which decreased the recovery. This may be due to the rotation velocity of the Falcon was not sufficient to hold dense particles. Increasing the G-force to 243 (70 Hz) increased the grade with higher recovery levels. The higher G-force was more suitable to hold higher amount of more dense particles. On the other hand, the grade was reduced with increasing water pressure due to escaping of more dense particles to the concentrate. The maximum grade of 38.46% with recovery of 85.4% was achieved at 70 Hz (equivalent to G-force 243) and 2 Psi. The best result of 38.46% Kerogen with 85.4% recovery was achieved at frequency of 70 Hz and water pressure of 2 Psi.

***Table 8.*** *Factors of Falcon concentrator tests.*

| Conditions | | Product | Weight% | Kerogen% | Recovery% |
|---|---|---|---|---|---|
| *F*(Hz) | Psi | | | | |
| 60 | 4 | Conc. | 93.8 | 33.85 | 96.3 |
| | | Tail | 6.2 | 20.58 | |
| | 5 | Conc. | 72.3 | 33.30 | 72.9 |
| | | Tail | 27.7 | 32.22 | |
| 70 | 2 | Conc. | 73.3 | 38.46 | 85.4 |
| | | Tail | 26.7 | 18.19 | |
| | 4 | Conc. | 88.9 | 33.67 | 90.7 |
| | | Tail | 11.1 | 27.82 | |
| | 5 | Conc. | 96.1 | 33.33 | 97.0 |
| | | Tail | 3.9 | 25.52 | |
| | | Head | 100 | 33.00 | 100 |

The coarse and fine concentrates were considered as a total concentrate. Table 9 shows that the total concentrate contained 39.66% kerogen with total recovery of 88.44%.

***Table 9.*** *The summary results of Falcon experiments.*

| Product | Weight% | Kerogen% | Recovery% |
|---|---|---|---|
| Coarse | 23.89 | 42.00 | 94.35 |
| Fine | 46.42 | 38.46 | 85.40 |
| Total | 70.31 | 39.66 | 88.44 |
| Feed | 100 | 31.50 | 100 |

## 4. Conclusions

Mineralogical analysis of the sample showed that the shale matrix is composed of lamina of 10 - 50 µm thickness. The lamina is consisted mainly of carbonate and argillaceous material that is rich inorganic matter. Quartz and pyrite were dispersed within the matrix. Phosphatic bioclasts including bone and scale remains of vertebrates and fishes were found. The sample is rich in carbon and oxygen as aliphatic and aromatic matrix. There are a high content of calcium oxide, sulfur, silica, alumina and iron oxide.

The maximum grinding was achieved at 90 min with 2: 1 solid/liquid ratio in presence of 12 rods. The lower and upper levels of both the centrifugal force and water pressure have been suggested to construct the design for the Falcon Concentrator type SB-40 to upgrade the coarse (−50 +25 µm) and fine (−25 µm) fractions.

It was found that higher frequency or G-force was needed to recover finer dense particles but may hinder coarser one. Increasing the G-force increased the grade while the recovery was increased with increasing water pressure. The grade was reduced at higher water pressure due to escaping of more dense particles to the concentrate. The coarse concentrate of 42% kerogen with 94.35% recovery was achieved at 60 Hz (equivalent to G-force 176) and water pressure of 4 Psi from feed of 29% kerogen. The fine concentrate of 38.4% kerogen with 85.4% recovery was achieved at 70 Hz (equivalent to G-force 243) and water pressure of 2 Psi from feed of 33% kerogen. The total concentrate of the coarse and fine concentrates grade was 39.66% kerogen with total recovery of 88.44%.

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