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# Chemical, Microscopic and XRD Studies on Makerwal Coal

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## ABSTRACT

The study is based on five representative samples of Makerwal coal which were collected from the T-Zeg-A Section of Makerwal mine during its operation. Samples were subjected to proximate analysis to determine the coal components. The significant constituents in the samples like fixed carbon content, sulphur, volatile matter and calorific values were respectively found in the range of 39.44-48.58%, 4.92-5.55%, 39.56-45.25% and 10156-11800 Btu/lb.

The detailed microscopic study of five polished sections of the samples was carried out which revealed that the coal samples contained mineral matter mainly in the form of shale, coarse and fine grained pyrite and specks of kaolinite. The vegetal matter was found in the form of good grade macerals. The X-ray diffraction (XRD) study confirmed the prescence of mineral constituents as indicated by the microscopic investigation. All the results so obtained were collectively analysed and concluded that the Makerwal coal belongs to a Subbituminous Coal Category. It was further confirmed by microscopic study that the mineral matter associated with the coal is largely separable and on its physical processing the grade of coal may be further improved.

## 1. INTRODUCTION

It is now an established fact that coal has derived from peat as a result of a number of metamorphic processes in various geological periods. Peat successively changed under influence of the overburden of sediments, heat and pressure and consequently a number of varieties of coal emerged. Some important varieties, referred as ranks, are now recognized almost universally in science and trade. The broad ranks of coal are: lignite coal, subbituminous coal, bituminous coal and anthracite coal. However, a number of subdivisions have also been reported in technical literature and commerce [1, 2].

Among the coal varieties, bituminous coal is considered to be a better quality due to its significance as a source for production of coke which is widely used as reducing agent in metallurgical processes. Other varieties such as lignite, subbituminous and anthracite have also vast applications as fuel in a number of industries like, power generation, cement, fertilizer, sugar, glass and ceramics and bricks firing, etc. Moreover, these are also used as fuel in locomotives, ships and serve as domestic heating source.

High and low grade coal deposits occur in a number of countries of the world. But unfortunately, Pakistan does not possess good quality coal. Its major coal resources are largely lignite and subbituminous [3].

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Earlier U.S. Geological Survey has decided to adopt the term subbituminous coal for the compact, so called black lignite, and to restrict the term lignite to the lower grade brown coal which ,on drying, split into slabs[4]. Subsequently, the US agencies formulated the following varied compositions of lignite and subbituminous coals which are still valid to a greater extent [5]. The following Table indicates the detailed composition of these varieties of coal.

**TABLE-1 PERCENTAGE COMPOSITION RANGES OF U.S. LIGNITE AND SUBBITUMINOUS COAL**

(Proximate and Ultimate Analyses)

| Components | Lignite coals (%) | Subbituminous coals (%) |
|---|---|---|
| Moisture | 8-43 | 2-41 |
| Volatile matter | 27-53 | 8-71 |
| Fixed carbon | 16-51 | 18-83 |
| Ash | 2.6-42 | 2-55 |
| Hydrogen | 5.14 | 1.76-6.98 |
| Carbon | 58.14 | 31-87 |
| Nitrogen | 1.05 | O.5-2 |
| Oxygen | 2.25-17 | 2.8-52.2 |
| Calorific value (Btu/lb) | 5,500-7000 | 6205-14843 |

Though the coal deposits in Pakistan are mostly of lignite rank, but there are some occurances where coal grade is closer to subbituminous coals. The Sharigh coal of Balochistan which, after washing, has been used by Pak Steel as a blend to the imported coal. Presently the Pak steel is procuring coals from other coal deposits like Chamlang (Balochistan). This indicates that there are prospects for utilizing indigenous coals in the local industrial units after these are upgraded to some extent discarding detrimental constituents like pyrite and excessive mineral matter.

Realizing these facts, a study on Makerwal coal has been planned to observe the nature of coal and closely examine the constituents and mineral phases of coal through proximate analysis, microscopic, X-ray diffraction (XRD) Spectroscopic techniques.

The microscopic examination of coal is quite common for coal evaluation, the first microscopic examination of coal was done by White [(6)] and his work was closely followed by many research workers. These earlier investigations, based on their microscopic examination on coal, placed the microscopic study on firm basis and gave evidence of their utility for coal research. Many other outstanding microscopists[(7)] have later on developed and perfected the technique of sectioning and polishing coal and have applied microscopic studies to the classification of constituents of coal until such studies have became a great scientific importance[(8).] One of the most important aspects of microscopic study is the coal petrography which is generally employed for the detailed study of macro and micro lithotypes. But for apparent identification of constituents of coal for the processing purposes, the microscopic knowledge of coal constituents is more than enough without particular regard to their source.

In addition the X-ray Diffraction (XRD) [(9)] is an efficient analytical technique used to identify and characterize unknown mineral matter in coals. Hence this technique provides excellent information regarding the mineral phases in coal which may be separated through coal processing techniques.

The present study, therefore, has been planned to closely examine the Makerwal coal belonging to New Incave T-zeg-A section through Proximate Analysis, Microscopy and XRD technique. This was considered to be essential to evaluate the physical nature of coal and its associated minerals for the processing point of view.

## 2. PLAN OF WORK

### 2.1 Makerwal Coal Mine and collection of samples

The area of Makerwal Collieries [(10)] is situated in the western escarpment of the famous salt range across the river Indus known as the Surghar range. These coal deposits were discovered at the end of $19^{th}$ century. Thus it is one of the oldest coal mines in Pakistan. The Surghar range consists entirely of sedimentary rocks ranging in age from Precambrian to recent.

The coal seams in the Makerwal area outcrop at an average height of 2300 ft above sea level while the main seams have overburden of upto 2500 ft thick. Geologically, the Makerwal coal belongs to the Eocene age.

The thickness of workable coal seam is generally 4 ft although it may, sometimes attain a thickness of 12 ft or thin down to 2 ft.The quality of Makerwal Coal appears to be subbituminous and considered to be quite good with calorific value of upto 12000 Btu’s.

Five representative samples of coal lumps, weighting 500 kg each, were statistically collected from new incave T-zeg- A section of Makerwal coal mine. The samples were tightly stored into plastic bags and subsequently used for investigations.

### 2.2-Sample Preparation

2.2.1- Sample for Microscopic Examination

From five sample bags (already referred) two lumps from each bag (2-4 inches in size) were picked up. Out of these ten lumps, five lumps of different appearance were selected for microscopic studies.

Polished specimens of coal are now widely employed in much the same manner as the metallographers use polished metals in their investigations [11]. The coal samples were cut manually and mounted on a solid base prepared by mixing Acrylic liquid and solid resins in the ratio of 2:3. The cold mounted samples were left for a while for setting. First grinding was done with a coarse abrasive (No.8 carborandum powder) followed by fine abrasives (200 alundum). Two successive polishing were carried out with the help of No. 600 carborandum. The specimens finally polished by slacked calcium oxide. The specimens were thoroughly washed and were dried under vaccum. The samples were than ready for microscopic examination.

2.2.2 Sample Preparation for Chemical Analysis and X-ray Spectroscopy

The remaining coal lumps in the five plastic bags were individually crushed by roll crusher and ground in a pebble mill in the size range of minus 100 mesh screen. The ground coal samples were systematically splitted into smaller samples weighing about 5g each using mechanical splitters. The samples were designated as A, B, C, D, E and saved for proximate analysis

Subsequently, smaller proportions of ground coal were taken from five bags and were thoroughly mixed to make a representative bulk sample and this sample was used for XRD investigation.

2.2.3- Experimental Work

The following investigations were finally carried out in the course of this study.

- Proximate Analysis
- Microscopic Studies.
- XRD Studies

## 3- EXPERIMENTAL PROCEDURES

### 3.1-Proximate Analysis

The Proximate analysis was carried out according to the ASTM Procedures as described in the literature [12].

### 3.2- Microscopic Study

The polished sections were examined on optical microscope at 100 magnification using reflected light source. The photomicrographs as appeared on Image Analyzer were taken as prints for detailed study of mineral constituents and various coal phases. Moreover, the structural features of coal samples such as Lithotypes and Microlithotypes were also studied to establish the grade of coal samples.

### 3.3- XRD Analysis

The powdered sample was split into two fractions (1- Unheated and 2- Heated at 550 $^oC$ for ½ hour) were analyzed at X’Pert Pro equipment by using powder diffractometeric technique and final results were obtained through X’Pert High Score plus software. The fractions were also examined through powder camera technique and the similar results were found.

## 4- RESULTS AND DISCUSSION

### 4.1-Proximate Analysis

Proximate Analysis of five coal samples is represented in the following Table:

**TABLE-2 PRIXIMATE ANALYSIS OF FIVE COAL SAMPLES**

| Sample | Moisture (%) | Volatile matter (%) | Ash (%) | Sulphur (%) | Fixed carbon (%) | Calorific value (kcal/kg) |
|---|---|---|---|---|---|---|
| A | 05.96 | 45.25 | 9.35 | 5.55 | 39.44 | 10325 |
| B | 4.05 | 40.05 | 08.19 | 05.00 | 47.71 | 11150 |
| C | 05.93 | 44.76 | 09.00 | 5.50 | 40.31 | 10156 |
| D | 04.00 | 39.99 | 08.00 | 04.97 | 48.01 | 11534 |
| E | 03.97 | 39.56 | 07.89 | 04.92 | 48.58 | 11800 |

The results indicate that the coal belongs to medium to high grade subbituminous rank. This is supported by international data available in literature (13). Moreover, the Calorific values of samples further support this conclusion.

### 4.2-Microscopic Analysis

SPACE FOR PHOTOMICROGRAPHS OF SAMPLES (A, B, C, D, E)

4.2.1-Photomicrograph of Coal Sample – A

The photomicrograph indicates some fibrous Lithotype Fusain (F) in the coal matrix making the coal sample belonging to a low grade subbituminous coal. The mineral matter as identified was mainly shale (S) with scattered pyrite (P) grains. The pyrite grains were also visible in the maceral phase. At the top left side of this photograph a resinite globule (R) was also observed which is one of the important feature of subbituminous coal. The findings regarding this photomicrograph were supported by a similar structure appeared in literature(14).

4.2.2-Photomicrograph of Coal Sample – B

The coal matrix exhibit various phases of macrinite (M), shale (S), vitrinite (V), prescence of kaolinite (K) in shale and the scattered particles of pyrite (P).The gangue minerals are present not only discretely but are embedded also in coal macerals. Similar photomicrograph may be seen in literature (15).

4.2.3-Photomicrograph of Coal Sample – C

The surface characteristics of photomicrograph reveal that the coal contains exinite (E), vitrinite (V), shale (S) and distributed pyrite (P) in lithotype and in gangue minerals. Moreover, some iron oxides (F) were also observed.

4.2.4-Photomicrograph of coal sample – D

Different phases identified in the photomicrograph were macrinite (M), fibrous fusain (F), pyrite grains (P) and mixture of shale (S), and kaolinite (K) embedded in fusain.

4.2.5-Photomicrograph of Coal Sample – E

This coal sample appears to be very high grade and is mainly composed of macrinite (M) and vitrinite (V) with very small proportions of gangue matter like shale (S), few grains of pyrite and traces of kaolinite (K) and iron oxide ($Fe_2O_3$).

**4.3- XRD ANALYSIS**

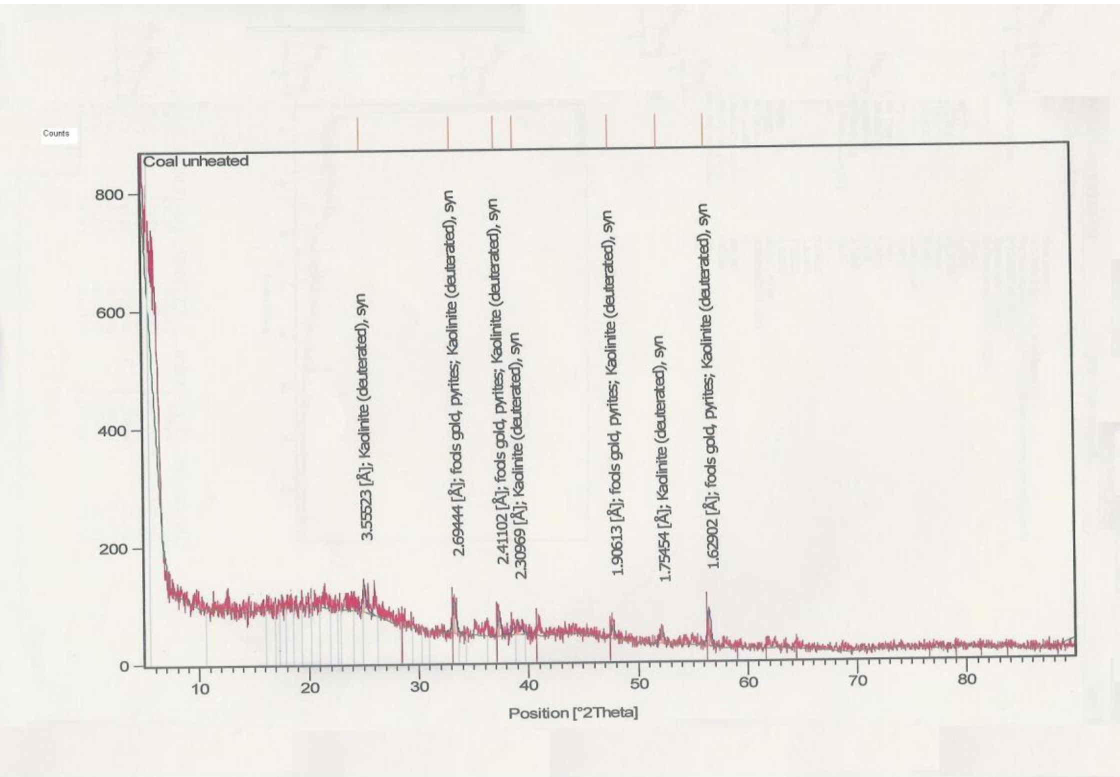


**FIG-1 XRD Graph of Bulk Coal Sample (Before Heating**)

**Peak List**

| **Pos. [2oTh.]** | **Height [cts]** | **FWHM [2oTh.]** | **d-spacing [Ao]** | **Rel. Int , [%]** |
|---|---|---|---|---|
| 25.0477 | 43.43 | 0.3542 | 3.55523 | 83.78 |
| 33.2518 | 51.84 | 0.3542 | 2.69444 | 100.00 |
| 37.2963 | 43.60 | 0.2362 | 2.41102 | 84.09 |
| 38.9972 | 14.18 | 0.9446 | 2.30969 | 27.35 |
| 47.7134 | 26.83 | 0.2362 | 1.90613 | 51.76 |
| 52.1306 | 25.00 | 0.3542 | 1.75454 | 48.23 |
| 56.4402 | 49.83 | 0.2880 | 1.62902 | 96.12 |

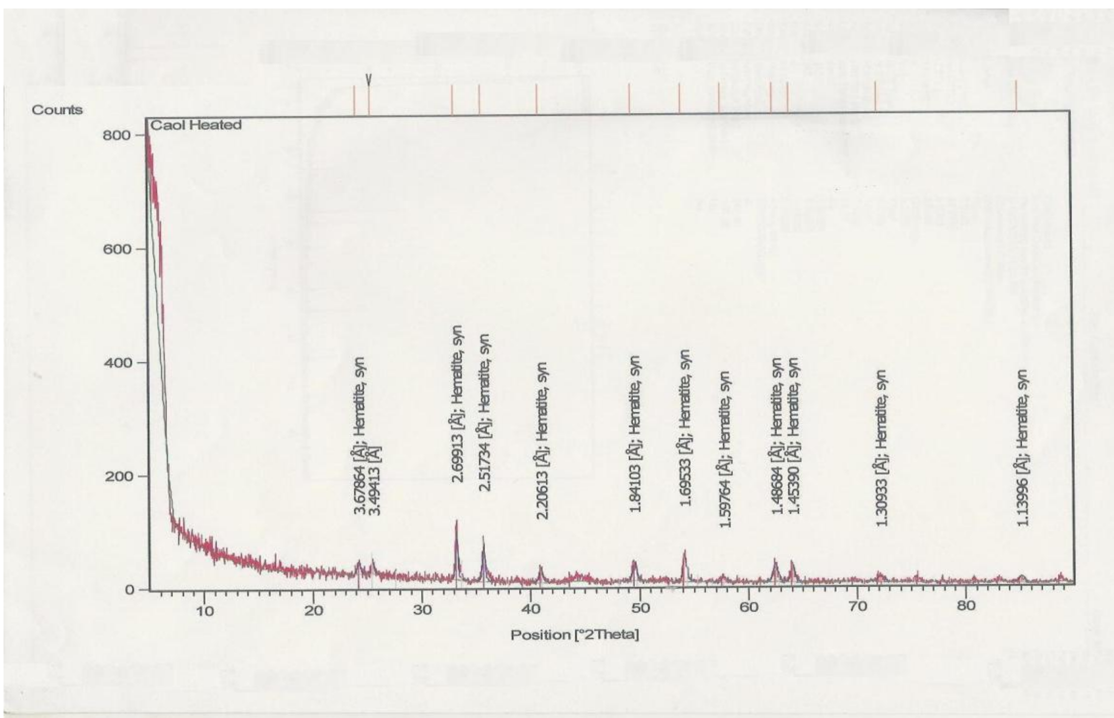


**FIG-2 XRD Graph of Bulk Coal Sample (After Heating at 550 oC)**

**Peak List**

| **Pos. [2$^o$Th.]** | **Height [cts]** | **FWHM [2$^o$Th.]** | **d-spacing [A$^o$]** | **Rel. Int , [%]** |
|---|---|---|---|---|
| 24.1944 | 19.49 | 0.3542 | 3.67864 | 23.29 |
| 25.4930 | 18.36 | 0.3542 | 3.49413 | 21.93 |
| 33.1923 | 83.70 | 0.2066 | 2.69913 | 100.00 |
| 35.6668 | 71.81 | 0.2362 | 2.51734 | 85.79 |
| 40.9077 | 23.53 | 0.3542 | 2.20613 | 28.11 |
| 49.5117 | 31.53 | 0.3542 | 1.84103 | 37.66 |
| 54.0965 | 43.62 | 0.3542 | 1.69533 | 52.12 |
| 57.7039 | 7.09 | 0.7085 | 1.59764 | 8.47 |
| 62.4643 | 30.37 | 0.4723 | 1.48684 | 36.29 |
| 64.0451 | 30.60 | 0.3542 | 1.45390 | 36.56 |
| 72.1443 | 8.79 | 0.7085 | 1.30933 | 10.50 |
| 85.0208 | 8.63 | 0.5760 | 1.13996 | 10.31 |

XRD Analysis of bulk coal sample is represented in the following Table:

TABLE-3 XRD ANALYSIS OF BULK COAL SAMPLE

| Crystalline minerals in coal matrix | Abundance |
|---|---|
| Kaolinite $(Al_2Si_2O_5)(OH)_4$ | It was identified as major clay mineral and was confirmed by heating the sample at 550oC for ½ hour. |
| Pyrite $(FeS_2)$ | It was present as a major phase of the crystalline mineral form in the coal matrix. |
| Hematite $(Fe_2O_3)$ | Its minor prescence was noted. |
| Quartz $(SiO_2)$ | Quartz as free mineral was not detected. |

The Graph of the unheated coal sample is presented in Fig-1. The data of peaks produced in peak list were investigated through the data cards and arrived at the conclusion that the sample contained pyrite, kaolinite and minor presence of hematite. It was further confirmed that the sample did not contain quartz.

A part of the same sample was heated at 550$^o$C for ½ hour in oxidizing atmosphere and the XRD Graph of the heated sample as presented in Fig.2 was obtained. The comparative examination of peaks on XRD graphs (unheated and heated) was made through the recorded peak lists. The peaks corresponding to pyrite in unheated sample appeared as the peaks of hematite in heated sample. The unaffected hematite appeared in both the unheated sample as well as in the heated sample. The peaks of kaolinite however, did not appear in the XRD pattern of heated sample. It is presumed that the kaolinite would have changed into amorphous form on heating that is why the kaolinite peak did not appear in the XRD graph.

## CONCLUSION

The outgoing discussion on the results which were obtained through proximate analysis, microscopic investigations and X-ray diffraction (XRD) study of Makerwal coal samples revealed that the coal belongs to subbituminous rank. It was further interpretted that the mineral matter in the coal existed largely in the separable form and its removal by physical processing techniques may further improve the grade of coal.


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