

A comparative XRPD study of Sedimentary Rocks of Pakistan

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ABSTRACT

The aim of this research project was to study the minerals/phases present in the rocky mountains of Attock, Pakistan. The phases/compounds present in the selected samples were identified by using hanawalt method and confirmed by computer programme "Rool, DQL Analysis" (Verification). The Compound Present in Rocks contained almost all the metallic, non metallic, categories of minerals including the sulphide group, the halide group, the oxide group, the corbonate group, the sulphate group, the phosphate group, along with organic minerals. The major identified phases along with their weight %age in descending order were found to be schauteite(25.97%), Albite(19.82%), illite(13.83%), ferrobustanite(7.55%), calcium led fluride (7.06%), Xonolite (6.74%), Gypsum(5.34%), Kaolonite(4.52%), Talc(2.69%), Dolomite(2.22%), Clinclore(2.08%), Hallite(1.50%) and Davadite(0.52%). These results are in accordance with the international results.

INTRODUCTION

A <u>rock</u> can be defined as a solid substance that occurs naturally because of the effects of three basic geological processes: <u>magma</u> solidification; <u>sedimentation</u> of <u>weathered</u> rock debris; and <u>metamorphism</u>.Rock, aggregation of solid matter composed of one or more of the <u>minerals</u> forming the earth's crust. The study of rocks is called petrology. Rocks are commonly divided, according to their origin, into three major classes-igneous, sedimentary, and metamorphic.

The solid part of the earth is made up of rocks. Rocks are made up of minerals, and minerals are composed of chemical elements. Minerals may be solid, liquids or gases the



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solid ones include minerals coals, metal ores, mineral salts, building material and any other minerals; liquid one's crude oil, brines, and mineral water as an example of gaseous minerals we may cite natural gases recovered through bore holes and subsequently burn or processed on the spot, are transported by pipe line over long distances. As a result of these processes, three main types of rock occur, Igneous Rocks, Sedimentary Rocks, Metamorphic Rocks, The most common igneous rocks are Rhyolite, Granite, Andesite, Diorite, Basalt, Gabbro, Peridotite, Obsidian, Pumice, Scoria, Igneous rocks are produced by solidification of molten magma from the mantle. Magma that solidifies at the Earth's surface conceives extrusive or volcanic igneous rocks. When magma cools and solidifies beneath the surface of the Earth intrusive or plutonic igneous rocks are formed. These form from hot, liquid rock 1500 °C, which cools. When this happens, crystals grow from the chemicals in the liquid. They form both from either Magma [inside the Earth], example Granite or of the Earth], example Rhyolite. These form from hot, liquid rock 1500 °C, which cools. When this happens, crystals grow from the chemicals in the liquid. Typical Felsic (light coloured) Igneous Rocks Granite & Rhyolite: Both of these contain similar chemicals [lots of Silicon, making an acid magma]. This makes them both light colored. Typical Mafic (dark coloured) Igneous rocks. Much like Rhyolite, small crystals - cooled quickly on the surface of the earth, so the crystals did not have much time to grow. Unlike Granite & Rhyolite, contain different chemicals [much less Silicon, making an alkaline magma]. This makes it very dark coloured. The most common sedimentary rocks, Shale, Sandstone, Conglomerate, Limestone, Tillite, Sedimentary Rocks, Sedimentary rocks are formed by burial, compression, and chemical modification of deposited weathered rock debris or sediments at the Earth's surface.Most of these are made from Sediment- bits of rock fragments, which have sunk to the bottom of rivers, or more usually the sea. They then get buried & squashed. Over time the fragments get stuck together to form rock. This happens because water also gets trapped with the sediment. Chemicals dissolved in the water make a form of 'glue, Chak & Limestone, however, are made from the shells of Dead Sea animals, Conglomerate Made from gravel, cemented together, Sandstone, Made from sand, cemented together, Shale, Made from mud, cemented together. Chalk Limestone Both of these are rich in Calcium carbonate [Ca CO₃]. This comes from the animal shells that these rocks contain. It is an alkali, and so these rocks effervesce - or fizz with dilute acid. Chalk is made from almost pure animal shells, and so tends to be white in colours. Limestone is a mixture of both animal shells and mud. This gives it its gray colour. The most common metamorphic



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rocks are <u>Slate</u>, <u>Phyllite</u>, <u>Schist</u>, <u>Gneiss</u>, <u>Marble</u>, <u>Quartzite</u> Metamorphic rocks are created when existing rock is chemically or physically modified by intense heat or pressure. Metamorphic rocks originate from the alteration of the texture and mineral constituents of igneous, sedimentary, and older metamorphic rocks under extreme heat and pressure deep within the earth. Some (e.g., <u>marble</u> and <u>quartzite</u>) are massive in structure; others, and particularly those which have been subject to the more extreme forms of metamorphism, are characterized by foliation (i.e., the arrangement of their minerals in roughly parallel planes, giving them a banded appearance). A distinguishing characteristic of many metamorphic rocks is their slaty cleavage</u>. Among the common metamorphic rocks are <u>schist</u> (e.g., mica schist and hornblende schist), <u>gneiss</u>, quartzite, <u>slate</u>, and marble.

MATERIALS AND METHODS:

MATERIALS:

SAMPLE PREPARATION:

The collected samples were strained with the help of muslin cloth to remove the fibrous and undesired straws etc. These strained samples were ground in order to obtain the fine powder and heated up to remove humidity because the humidity affects the result very badly.

These samples were filled in a rectangular hole of a rectangular disc by pressing it gently with the help of smooth mirror slit. One side of the sample holder was provided with a small dot indicating the front of the slit. The front side of the sample holder was placed on the mirror slit and the hole was filled with the powder sample and then pressed it gently with an other mirror slit, until the whole was completely filled with the powder and the surface of the powder was exactly in line with the surface of the sample holder.



The extra powder was removed. This method was repeated for all the other samples. The sample holder was washed with the acetone, cleaned and dried before filling each sample.

PRINCIPLE OF X-RAY DIFFRACTOMETER:

X-ray diffractometre works on the principle that when sample is irradiated by the x-rays, is diffracted in such a way that is characteristic of compound present in the sample.

X-RAY POWDER DIFFRACTION METHOD:

(X-Ray powder diffraction (XRPD) method has proved very useful in study of atmospheric aerosols Ballantyne, C.K.. 1994, Baron, Dirk, Palmer, D- Card, Stanley and T. James. 1996, Cullity. B.D. 1978, Davis, B.L,1989, Esteve, V.J., L.E. Rius and J.M. Amigo. 1997, Fanziah, L., S. Zanyah and T. Jamal. 1996, Fayyaz, Q.M., 2001 etc.). In this technique the crystal to be examined must be in the form of fine powder. The incident monochromatic radiation strikes the finely ground powder specimen contained a thin walled capillary tube held at a certain angle to the incident X-Ray beam. Only those planes diffract the X-Ray for which the Bragg's law is satisfied. Hence, X-ray powder diffraction (XRPD) method is of course the only method that can be used when a single crystal is not available. It is specially suited for the determining the lattice parameters with high precision and for the identification of phases.

MATRIX FLUSHING METHOD:

This method provides the exact relationship between intensity and concentration free from matrix effects. This method is very useful because amount of amorphous phase content present in the other crystalline phases can also be detected. The maximum error is quantifying a phase in a mixture by the matrix flushing method had been estimated to be 80% relative. This method was applicable when all phases in the mixtures were in crystalline form. In this method, a fundamental matrix flushing concept was introduced. Let xi be the weight fraction of a component "i" in the mixture of "n" components then basic intensity equation could be written as:

 $Ii = KiXi \qquad \dots \qquad (1)$

Where K is a constant.



For quantitative analysis of mixture of n components the above equation became a matrix equation:

[I] = [KX].....(2)

The equation had a unique solution if the rank of K was equal to the rank of the (K, 1) matrix. The equation will be of the form:

 $Xi = [Ki/Ii (\sum_{j=1}^{n} Ij/Kj)]^{-1} x100....(3)$

The above relation gave the percentage composition of a component i in mixture of n componets. In equation (3) Ii is integrated intensity and Ki is relative intensity ratio given by

 $Ki = [Ij/I_{kcl}]_{50/50}$

This ratio can be calculated by mixing the component i with the standard material KCl in the ratio 1:1. The relative intensity calculated for the seven minerals are shown in a table:

In the present study, quantitative phase analysis of solid aerosols was carried out by powder x-ray diffraction method to identify the phase of the compounds.

QUALITATIVE PHASE ANALYSIS:

Each crystalline material gives a unique X-ray diffraction pattern. Qualitative phase analysis was used for study of crystal structure and unknown phases of material. In XRPD pattern there were two parameters (Bragg's angle and integrated intensities). Bragg's equation is used to find the d-value corresponding to Bragg's angles. The dvalues which were obtained from samples were compared with standard values. This was done by employing the Joint Committee Powder Diffraction Standard (JCPDS) file



method. With the help of JCPDS, the existence of different minerals in the sample was confirmed.

THE HANAWALT METHOD:

The principle of identification of substances by powder x-ray diffraction is based on the fact that every crystalline material gives its own characteristics pattern. This pattern of material in a mixture form is independent of others. The powder data of crystalline material is now published by ICDD (International Center for Diffraction Data) in the form of cards. Each card contains the name of material studied by the powder x-ray diffraction and corresponding miller planes of reflections belong to the materials. A search manual (index book) is also published by the ICDD which contains maximum intensity reflections of all the identified crystalline materials.

RESULTS AND DISCUSSION

 Table 2K01: Identified Phases for Rock Sample-1

| Peak No. | 20(Degree) | d- | Cnc | Wt/ % | Identified |
|----------|------------|------------------------|-------|-------|------------|
| Peak NO. | 2θ(Degree) | value(A ⁰) | Cps | age | Phases |
| 1 | 6.251 | 14.1285 | 7.01 | 3.10 | Cl |
| 2 | 8.854 | 9.9787 | 20.17 | 8.92 | ILL |
| 3 | 10.500 | 8.4183 | 18.83 | 8.33 | N.D |
| 4 | 12.507 | 7.0713 | 12.44 | 5.50 | Kn |
| 5 | 13.835 | 6.3954 | 11.16 | 4.94 | N.D |



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| 6 | 15.101 | 5.8622 | 8.75 | 3.87 | N.D |
|----|--------|--------|--------|--------|-------------|
| | | | | | |
| 7 | 17.802 | 4.9784 | 6.49 | 2.87 | ILL |
| 8 | 18.883 | 4.6958 | 7.06 | 3.12 | N.D |
| 9 | 19.711 | 4.5003 | 5.05 | 2.23 | ILL |
| 10 | 20.887 | 4.2495 | 41.60 | 18.39 | QZ |
| 11 | 22.062 | 4.0257 | 17.95 | 7.94 | N.D |
| 12 | 23.576 | 3.7705 | 11.22 | 4.96 | N.D |
| 13 | 25.182 | 3.5336 | 8.65 | 3.82 | Cl |
| 14 | 25.566 | 3.4814 | 12.83 | 5.67 | Kn |
| 15 | 26.668 | 3.3399 | 226.18 | 100.00 | Sch, ILL |
| 16 | 27.521 | 3.2383 | 10.58 | 4.68 | ILL |
| 17 | 27.951 | 3.1895 | 26.58 | 11.75 | Cl, Fr |
| 18 | 29.501 | 3.0254 | 22.39 | 9.90 | Sch |
| 19 | 31.899 | 2.8031 | 7.90 | 3.49 | ILL |
| 20 | 33.618 | 2.6636 | 6.06 | 2.68 | Da, Cl, CLF |
| 21 | 36.552 | 2.4563 | 13.44 | 5.94 | QZ |
| 22 | 39.509 | 2.2790 | 23.68 | 10.47 | Cl, Fr, |
| 23 | 40.329 | 2.2345 | 5.51 | 2.44 | ILL |
| 24 | 42.466 | 2.1269 | 12.11 | 5.36 | Gyp |
| 25 | 50.127 | 1.8183 | 16.34 | 7.22 | Sch |



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| 26 | 54.887 | 1.6714 | 8.35 | 3.69 | Da |
|----|--------|--------|------|------|----|
| 27 | 55.352 | 1.6584 | 5.25 | 2.32 | Cl |

| Deek Ne | 20(Decree) | d- | Cine | Wt/ % | Identified |
|----------|------------|------------------------|-------|-------|--------------|
| Peak No. | 2θ(Degree) | value(A ⁰) | Cps | age | Phases |
| 1 | 6.251 | 14.1285 | 5.92 | 2.33 | Т |
| 2 | 8.854 | 9.9775 | 13.78 | 5.43 | ILL |
| 3 | 10.557 | 8.3725 | 7.30 | 2.87 | |
| 4 | 12.442 | 7.1081 | 11.25 | 4.43 | CLF |
| 5 | 13.932 | 6.3512 | 8.52 | 3.36 | Xn |
| 6 | 17.746 | 4.9939 | 8.82 | 3.48 | |
| 7 | 19.832 | 4.4731 | 10.77 | 4.24 | |
| 8 | 20.843 | 4.2583 | 44.84 | 17.66 | CLF, Sch, Xn |
| 9 | 20.005 | 4.0361 | 12.23 | 4.82 | Do |
| 10 | 22.925 | 3.8760 | 4.96 | 1.96 | Cl 22 |
| 11 | 25.665 | 3.4682 | 6.25 | 2.46 | |



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| 12 | 26.621 | 3.3457 | 253.87 | 100.00 | Sch, ILL |
|----|--------|--------|--------|--------|-------------|
| 13 | 27.921 | 3.1928 | 28.52 | 11.23 | Fr, ILL |
| 14 | 30.393 | 2.9385 | 6.29 | 2.48 | |
| 15 | 31.209 | 2.8636 | 7.64 | 3.01 | Da, Gyp |
| 16 | 35.940 | 2.4967 | 7.26 | 2.86 | Gyp |
| 17 | 36.552 | 2.4563 | 18.65 | 7.35 | CLF, K |
| 18 | 39.447 | 2.2824 | 10.76 | 4.24 | Cl, Fr |
| 19 | 40.242 | 2.2392 | 8.09 | 3.19 | Gyp |
| 20 | 42.484 | 2.1260 | 15.35 | 6.05 | Cl, Fr |
| 21 | 45.937 | 1.9798 | 10.80 | 4.26 | Н, Gyp |
| 22 | 50.138 | 1.8179 | 45.11 | 17.77 | ILL, Gyp, K |
| 23 | 56.496 | 1.6275 | 4.19 | 1.65 | |

 Table 2K02: Identified Phases for Rock Sample-2



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| Peak No. | 2θ(Degree) | d- value(A ⁰) | Cps | Wt/% age | Identified Phases |
|----------|------------|------------------------------|-------|-------------|----------------------|
| 1 | 5.753 | 15.3500 | 14.45 | 6.61 | |
| 2 | 6.226 | 14.1848 | 10.21 | 4.67 | Т |
| 3 | 8.864 | 9.9683 | 25.45 | 11.65 | ILL |
| 4 | 12.482 | 7.0858 | 13.09 | 5.99 | Cl |
| 5 | 13.070 | 6.7684 | 9.08 | 4.15 | Kn |
| 6 | 15.995 | 5.5366 | 6.29 | 2.88 | |
| 7 | 17.687 | 5.0105 | 5.95 | 2.72 | ILL |
| 8 | 19.370 | 4.5788 | 6.51 | 2.98 | T, ILL |
| 9 | 19.897 | 4.4586 | 8.51 | 3.90 | ILL |
| 10 | 20.861 | 4.2547 | 68.81 | 31.49 | Xn, CLF, Sch |
| 11 | 22.029 | 4.0316 | 8.82 | 4.04 | Do |
| 12 | 22.989 | 3.8655 | 6.71 | 3.07 | Cl |
| 13 | 23.582 | 3.7695 | 17.06 | 7.81 | Xn |
| 14 | 25.152 | 3.5377 | 13.32 | 6.10 | K ²⁴ |
| 15 | 25.481 | 3.4928 | 12.08 | 5.53 | |



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| 16 | 26.643 | 3.3430 | 218.52 | 100.00 | Sch, ILL |
|----|--------|--------|--------|--------|----------------|
| 17 | 27.962 | 3.1882 | 86.56 | 39.61 | Al, Gyp |
| 18 | 29.456 | 3.0299 | 10.60 | 4.85 | Cl, Fr |
| 19 | 32.756 | 2.7318 | 5.36 | 2.45 | Da |
| 20 | 35.040 | 2.5587 | 10.07 | 4.61 | Cl |
| 21 | 36.541 | 2.4570 | 14.55 | 6.66 | Gyp, CLF, K |
| 22 | 39.479 | 2.2806 | 24.59 | 11.25 | Cl, Fr |
| 23 | 40.349 | 2.2334 | 13.09 | 5.99 | Gyp |
| 24 | 42.463 | 2.1270 | 18.68 | 8.55 | Sch |
| 25 | 47.284 | 1.9208 | 5.30 | 2.43 | Da, Cl, CLF, K |
| 26 | 50.159 | 1.8172 | 34.17 | 15.64 | Gyp, К |
| 27 | 52.368 | 1.7456 | 3.90 | 1.79 | |

Table 2K03: Identified Phases for Rock Sample-3.

| DealsNe | 20 | d volvo | | 0/ | Phase 25 |
|----------|----|---------|-----|----|----------------|
| Peak No. | 20 | d-value | Cps | % | Identification |
| | | | | | |



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| 1 | 5.962 | 14.8117 | 17.90 | 7.15 | CL |
|----|--------|---------|--------|--------|--------------|
| 2 | 8.835 | 10.0001 | 24.31 | 9.72 | ILL |
| 3 | 12.491 | 7.0806 | 8.46 | 3.38 | Kn |
| 4 | 13.778 | 6.4217 | 5.99 | 9.39 | |
| 5 | 17.798 | 4.9793 | 6.93 | 2.77 | T, ILL |
| 6 | 20.845 | 4.2579 | 54.55 | 21.80 | Xn, CLF, Sch |
| 7 | 22.989 | 3.8654 | 8.22 | 3.29 | Cl |
| 8 | 26.646 | 3.3426 | 250.16 | 100.00 | Sch, ILL |
| 9 | 28.139 | 3.1685 | 30.54 | 12.21 | Cl, Fr |
| 10 | 29.707 | 3.0048 | 18.00 | 7.19 | CAL |
| 11 | 31.289 | 2.8564 | 5.70 | 2.28 | ILL |
| 12 | 34.907 | 2.5682 | 8.01 | 3.20 | ILL |
| 13 | 36.560 | 2.4558 | 20.86 | 8.34 | QZ |
| 14 | 39.465 | 2.2814 | 17.09 | 6.83 | QZ |
| 15 | 40.295 | 2.2363 | 9.28 | 3.71 | Cl |
| 16 | 42.449 | 2.1277 | 20.95 | 8.37 | QZ |



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| 17 | 45.457 | 1.9937 | 10.00 | 4.00 | QZ |
|----|--------|--------|-------|------|-------|
| 18 | 45.803 | 1.9794 | 11.02 | 4.41 | QZ |
| 19 | 46.647 | 1.9455 | 4.32 | 1.73 | ILL |
| 20 | 49.273 | 1.8478 | 4.67 | 1.87 | QZ |
| 21 | 50.162 | 1.8171 | 19.99 | 7.99 | Xn, K |
| 22 | 54.879 | 1.6716 | 10.34 | 4.13 | QZ |

Table 2K04: Identified Phases for Rock Sample-4.

Table 2K05:Weight %age of Identified Phases

| Phases | Weight / %age |
|------------|---------------|
| Talc | 2.69 |
| Schauteite | 25.97 |



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| Illite | 13.83 |
|-----------------------|-------|
| Albite | 19.82 |
| Chlinclore | 2.08 |
| Gypsum | 5.34 |
| Calcium Lead Fluoride | 7.06 |
| Xonolite | 6.74 |
| Dolomite | 2.22 |
| Davadite | 0.52 |
| Kaolonite | 4.52 |
| Ferrobustamite | 7.55 |
| Hallite | 1.50 |
| | |

Table 2K06:Quantitative Phase Analysis of the Identified Phases from KiranaHills (Attique. 1995-1997 M.Phil Thesis).

| Phase | K01 | K03 | K05 | K07 | K08 |
|-------|-----|-----|-----|-----|-----|
| | | | | | |



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| А | 60% | N.D | 9% | N.D | 24%, 10% |
|----------|-----|-----|-----|-----|----------|
| G/IL | 16% | 17% | 10% | 05% | 08% |
| Q/SODD | 12% | 13% | N.D | 30% | N.D |
| A/CA/Zr | 08% | 10% | N.D | 04% | 08% |
| XEN/Cart | 04% | N.D | 12% | N.D | N.D |
| SODD | N.D | 50% | 60% | 50% | 16% |
| Cart | N.D | 19% | 09% | N.D | N.D |
| Th | N.D | N.D | N.D | 05% | 09% |
| Q/Cart | N.D | N.D | N.D | 06% | 07% |
| СН | N.D | N.D | N.D | N.D | 18% |

The phases/compounds present in the selected samples were identified by using hanawalt method and confirmed by computer programme "Rool, DQL Analysis" (Verification). The diffraction pattern was obtained in the range of 2θ this analysis showed the presence of Schaurteitc, Gypsum, Calcite, Calcium Lead Fluoride, Halite, Clinchlore, Dolomite, Davadite, Ferrobustamite, Xonotlite, Kaolonite, Albite, Talc, Quartz, Dolomite and Illite. In all these samples Schauteite, Calcium Lead Flouride, Xonotlite, Illite, Ferrobustamite, Talc, Gypsum, Pottasium major minerals and Hallite, Dolomite, Clinchlore, Davadite, Kaolonite, Calcite, Albite were dominant minerals present in the rocky mountains of Attock. The quantitative analysis of the identified phases was carried out by matrix flushing method. The Compound Present in Rocks contained almost all the metallic, non metallic, categories of minerals including the sulphide group, the halide group, the oxide group, the corbonate group, the sulphate group, the phosphate group, along with organic minerals. The major identified phases along with their weight % age in descending order were found to be schauteite(25.97%), Albite(19.82%), illite(13.83%), ferrobustanite(7.55%), calcium led fluride (7.06%), Xonolite (6.74%), Gypsum(5.34%), Kaolonite(4.52%), Talc(2.69%), Dolomite(2.22%), Clinclore(2.08%), Hallite(1.50%) and Davadite(0.52%). These results are in accordance with the results of Schroeder, P.A. and J Shiflet. 2000, Serge, J, S.P, Idefonse and M. Bouncharel, 2000, Wang WM, Yeh H.W.,



P.Y. Chen, MK Wang. 1998 etc and slightly disagree with the findings of Bhatti, T.M. et al, Boliver, J.P. et al, Gosh, A et al, Hamid B.N., M et al. Hussein, A et al.

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