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Quality change using random mixing of soils from different areas of Punjab by means of XRD technique

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The results obtained from investigation of soil samples collected from different units of Punjab like agricultural, commercial and industrial units are reported and analyzed in this paper. XRPD studies from various units showed that the phases present in the soil samples were Quartz (SiO₂), Calcite (CaCO₃), Albite NaAlSi₃O₄, Chlorophane Mg₅Al(Si,Al)₄O₁₀, Illite {(K,H₃O) Al₂Si₃AlO₁₀(OH)₂}, Vermiculite {Mg(Mg,Fe)₃(Si,Al)₄O₁₀(OH)₂.4H₂O}, Gypsum (CaSO₄.2H₂O), Talcum {Mg₃Si₄O₁₀(OH)₂}, Aluminate Al₂O₃, Chamosite (Fe,Al,Mg,Mn)₆(Si,Al)₄O₁₀(OH)₈, Dolomite (Ca,Mg)CO₃ and Epidote Ca₂(Al,Fe)₃(SiO₂O₇)(SiO₄)(OH)₂. The result and mean values of concentrated data in weight %age of phases showed that Quartz, Illite, Albite and Calcite were the major constituents of the soil of Narowal and Faisalabad, while Quartz, Illite and Chamosite were the major constituents of soil in Multan. But, all other phases appeared as minor components of the soil. These were present in very small amount in the soil. From the plot of the comparison of the mean weight percentage of phases, it was clear that Illite was major phase in largest amount (53.27%, 42.62%) in the soil of Faisalabad and Narowal respectively, while Quartz (SiO₂) appeared as the major phase in the largest amount (68.83%) in the soil of Multan. The special sample S-1 showed that some minerals which were already present in the soil of Narowal and Faisalabad disappeared after combining the soil samples. It means that there was a chemical phase which not only became the reason for disappearance of the minor phases but also affected the values of major phases. The appeared phases were Q, CAL and ILL while the disappeared phases were AL, CL, G, T and AM. In the same way, in the special sample S-2, CH, DL and EP did not appear after combining all the samples of Multan.

Key words: XRPD phase analysis, sample mixing, readjustment of the phases, weight %age concentration, soil fertility upgradation, area, specific mineralogy.

INTRODUCTION

The term soil is derived from a Latin word "Solum" which means floor. It is defined in Webster's collegiate dictionary as "the upper layer of the earth which may be dug, ploughed, etc., especially the loose surface material of the earth in which plant grow". This is a very simple definition for a very complex material. The soil contains small but significant quantities of organic and inorganic compounds, some of which contain elements, which are essential for plant growth. Collection of natural bodies

occupying the portion of earth that support plants or can support plants out of door have properties due to integrated effect of climate, living matter. All these things acting upon the parent material and the effect of parent material are modified by relief and topography, and all these conditions are affected by time (Adam et al., 1983; Aroki and Khoyame, 1998; Baver, 1956; Burnett, 1995; Bish and Aronson, 1993). Soil is complex because of the extreme variability in Physical and Chemical composition. They are formed from exposed masses of partially weathered rocks and minerals with different chemical composition, different degree of resistance to weathering and different physical properties. Other complete ways of describing soils are mixture of minerals and organic

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matter that are capable of supporting plant life. Minerals make up about 50% of the volume of most soils. They provide physical support for plants and create the water and air-filled pores that make plant growth possible. Most of the elements in the crust and in soil occur in minerals. A mineral is defined as a naturally occurring homogeneous solid with a definite (but not generally fixed) chemical composition and an ordered atomic arrangement, and is usually formed by inorganic processes (Ron et al., 1999; Russell, 1957). Both chemical composition and crystal structure (ordered atomic arrangement) are important parts of this definition; neither alone is sufficient to explain the properties of minerals. Soil minerals also are referred to as either primary or secondary minerals. The primary and secondary mineral classes are not mutually exclusive, and some minerals occur in both. There are twelve elements that are essential to plant growth, which are provided by soil. These twelve elements consist of six macro elements: Calcium (Ca), Magnesium (Mg), Potassium (K), Nitrogen (N), Phosphorous (P) and Sulfur (S), and six micro elements: Copper (Cu), Manganese (Mn), Iron (Fe), Zinc (Zn), Boron (B) and Molybdenum (Mo). These twelve elements along with Carbon (C), Hydrogen (H) and Oxygen (O) make up 15 essential elements. Soil pH indicates how acid ($\text{pH} < 7$, high H^+ concentration) or basic ($\text{pH} > 7$, high OH^- concentration) is the soil solution. Soil pH is influenced by parent soil materials and tends to decrease with time. Soils with low base (Ca, Mg, K, etc.) status, such as those in the South Eastern United States, are sensitive to the acidifying effects of Nitrogen fertilizers (including organic N sources). Addition of lime stone and other basic material is normally used to maintain soil pH in a desirable range. Although organic matter additions did not directly affect soil pH, soils that receive significant amount of organic material tend to maintain soil pH value for longer periods of time. Most crops grow in the temperate mid-Atlantic, though South Eastern United States prefers slightly acidic soils (that is, pH of 5.6-6.8). Lower or higher pH value can cause plant nutrient deficiencies. These results were also compared with those of other similar studies quoted in national and international journals having impact factor. This effort is the extension of our PhD project; most of the work have been presented and published elsewhere (Marsonia et al., 2008; Meena et al., 2011). The objectives of this study are to access the soil fertility, quality change using random mixing of soils from different areas of Punjab by means of XRD technique and make recommendations of the same technique used by soil experts in parks and gardens for the farmers (Hughes, 1990; Imam, 1992; Jones and Malik, 1990).

MATERIALS AND METHODS

Site selection

A total of 15 samples of soil from five different locations

of each unit (Agricultural, Commercial and Industrial Units of the Punjab) were randomly collected. The description of the selected observational sites of the Punjab is given in Table 1.

Sample collection

The samples were collected at Faisalabad, Narowal and Multan at a depth of 0-3 inches. The samples were packed in small plastic bags and then labeled for particular sample sites. Two special samples were also made from the aforementioned 15 selected samples by the following way:

Special sample no. 1 (S-1): It was made by combining all the samples selected from Faisalabad and Narowal.

Special sample no. 2 (S-2): It was made by combining all the samples selected from Multan.

Sample preparation

The collected samples were strained to remove the fibrous and undesired materials with the help of muslin cloth and dried by heating to remove humidity because humidity affects the results very badly and then ground to make them homogeneous before XRPD analysis.

Sample loading

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 gently with another mirror slit, until the hole 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.

Qualitative phase analysis (Hanawalt method)

Each crystalline material gives a unique X-ray diffraction pattern. Qualitative phase analysis was used for the 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 was used to find the d-value corresponding to Bragg's angles. The d-values 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 (Shahid and Hussain, 2004, 2007);

Table 1. Comprehensive description of selected sites of Punjab.

Name of the city	Type of the site	Selected sites	Site description
Narowal	Agricultural Unit	Hussaini chowk (NC-1)	19° N-E at 1.3Km
		Tehsil chowk (NC-2)	57° W-S at 1.4Km
		Zafarwal chowk (NC-3)	14° N-E at 1.9Km
		Phatak chowk (NC-4)	25° N-W at 2.2 Km
		Siddiqia chowk (NC-5)	at Origin
Multan	Commercial Unit	Dulat gate chowk (MC-1)	11° E-S at 1.3Km
		Clock Tower chowk (MC-2)	at Origin
		Daira Adda chowk (MC-3)	37° W-S at 2.4 Km
		Chowk Shahidan (MC-4)	15° N-E at 1.2 Km
		General Bus stand chowk	25° E-S at 5.7 Km
Faisalabad	Industrial Unit	Millat chowk (FC-1)	5° N-E at 12.9Km
		D-Type chowk (FC-2)	32° S-E at 1.5 Km
		Billal chowk (FC-3)	at Origin
		Chenab chowk (FC-4)	21° N-W at 5Km
		Babar chowk (FC-5)	57° N-E at 5.5Km

Shahid et al., 2008, 2009, 2012¹; Shahid and Nasim, 2009).

Quantitative phase analysis (Matrix flushing method)

This method provides the exact relationship between intensity and concentration free from matrix effects. It is very useful because the amount of amorphous phase content present in the other crystalline phases can also be detected. The maximum error in 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 mixture were in crystalline form. In this method, a fundamental matrix flushing concept was introduced. Let x_i be the weight fraction of a component "i" in the mixture of "n" components, then basic intensity equation could be written as:

$$I_i = K X_i \quad \dots\dots\dots (1)$$

where K is a constant.

For quantitative analysis of mixture of n components, equation 1 becomes a matrix equation:

$$[I] = [KX] \quad \dots\dots\dots (2)$$

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:

$$X_i = [K_i / I_i (\sum_{j=1}^n I_j / K_j)]^{-1} \times 100 \quad \dots\dots\dots (3)$$

The above relation gave the percentage composition of

component i in the mixture of n components. In equation (3), I_i is the integrated intensity and K_i is the relative intensity ratio given by $K_i = [I_j / I_{KCl}]_{50/50}$.

This ratio can be calculated by mixing the component i with the standard material KCl in the ratio 1:1 (Dengh, 1996; Esteve, 1997; Fejital, 1989; Farmer, 1974; Ghosh, 2002). The relative intensity calculated for the seven minerals are shown in Table 2.

In the present study, quantitative phase analysis of soil samples was carried out by powder x-ray diffraction method to identify the phase of the compounds (Jumikis, 1989; Kittle, 1976; Steel and Torrie, 1997; Toor, 1998; Wang et al., 1998; Shahid, 2010).

RESULTS AND DISCUSSION

The phases or minerals present in selected samples were identified using computer programming. The phases present in the soil samples were Quartz (SiO_2), Calcite (CaCO_3), Albite $\text{NaAlSi}_3\text{O}_8$, Chlinochlore $\text{Mg}_5\text{Al}(\text{Si},\text{Al})_4\text{O}_{10}$, Illite $\{(\text{K},\text{H}_3\text{O}) \text{Al}_2\text{Si}_3\text{AlO}_{10}(\text{OH})_2$, Vermiculite $\{\text{Mg}(\text{Mg},\text{Fe})_3 (\text{Si},\text{Al})_4 \text{O}_{10}(\text{OH})_2 \cdot 4\text{H}_2\text{O}\}$, Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), Talcum $\{\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2\}$, Aluminate Al_2O_3 , Chamosite $(\text{Fe},\text{Al},\text{Mg},\text{Mn})_6 (\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_8$, Dolomite $(\text{Ca},\text{Mg})\text{CO}_3$ and Epidote $\text{Ca}_2(\text{Al},\text{Fe})_3 (\text{SiO}_2\text{O}_7) (\text{SiO}_4)(\text{OH})_2$. Tables 3 to 8 describing the mean values of concentrated data in weight percentage of phases explain that Quartz, Illite, Albite and Calcite were the major constituents of the soil of Narowal and Faisalabd, while Quartz, Illite and Chamosite were the major constituents of the soil of Multan. But all other phases appeared as minor components of the soil. These were present in very small

Table 2. Relative intensity of soil samples with KCl.

Compound phases/ Minerals	Relative intensity $K_i = I_i/I_{KCl}$
Quartz	0.85
Illite	0.20
Talc	0.32
Gypsum	0.83
Chlorite/Chlinochlore	0.23
Albite	0.36
Calcite	0.74

Table 3. Concentration data in Mean Weight percentages of the phases present in soil of Commercial, Industrial and Agricultural Units of the big city of the Punjab.

Phases identified	Mean Weight percentages of identified phases from the selected cities		
	Multan	Narowal	Faisalabad
ILL	14.14	42.62	53.27
Q	68.83	14.95	18.25
CAL	3.80	16.83	11.65
AL	BDL	10.90	13.31
CL	BDL	4.43	2.22
VER	1.58	BDL	BDL
G	BDL	6.97	s
T	BDL	2.69	BDL
AM	BDL	0.61	BDL
CH	10.26	BDL	BDL
DL	1.10	BDL	BDL
EP	0.26	BDL	BDL

Table 4. XRPD analysis of Special sample (S-1) of soil to identify the various phases.

Peak No	2 θ	d-values	FWHM	Phases Identified
1	20.781	4.27455	0.590	Q
2	26.589	3.35258	0.590	Q
3	27.884	3.19972	0.394	ILL
4	29.467	3.03129	0.590	CAL
5	36.465	2.46404	0.590	ILL
6	43.463	2.08218	0.394	Q
7	45.606	1.98918	0.590	ILL
8	50.124	1.81996	0.590	Q
9	54.94	1.67147	0.590	Q
10	59.94	1.54341	0.590	Q
11	63.891	1.45705	0.590	Q
12	68.037	1.37800	0.787	Q
13	75.702	1.25639	0.590	Q
14	77.601	1.23032	0.590	Q
15	81.400	1.18224	0.590	BDL
16	83.818	1.15418	0.590	BDL
17	90.844	1.08232	0.590	BDL
18	94.859	1.04683	0.787	BDL
19	102.351	0.98956	0.787	BDL.
20	114.483	0.91598	0.960	BDL

Table 5. Weight percentages of phases present in Special sample (S-1).

Phases identified	Integrated Intensity (I _i)	Weight percentage
Q	1298.9	77.64
AL	BDL	BDL
ILL	337.5	20.17
CAL	36.6	2.19
CL	BDL	BDL
T	BDL	BDL
G	BDL	BDL
CH	BDL	BDL
AM	BDL	BDL
EP	BDL	BDL
DL	BDL	BDL
VERM	BDL	BDL

Table 6. XRPD analysis of Special sample (S-2) of soil to identify the various phases.

Peak No	2 θ	d-values	FWHM	Identified phases
1	20.778	4.27521	0.590	Q
2	26.952	3.35218	0.394	Q
3	27.892	3.19878	0.590	ILL
4	29.473	3.03073	0.590	CAL
5	34.741	2.58229	0.590	VERM
6	39.376	2.28835	0.590	CAL
7	43.438	2.08329	0.590	Q
8	45.582	1.99018	0.590	Q
9	50.112	1.82036	0.787	ILL
10	54.906	1.67224	0.590	Q
11	68.071	1.37739	0.787	Q
12	79.837	1.20141	0.590	BDL
13	81.400	1.18224	0.590	BDL
14	83.806	1.15432	0.590	BDL
15	90.871	1.08207	0.590	BDL
16	106.642	0.96127	0.787	BDL
17	114.506	0.91586	0.960	BDL

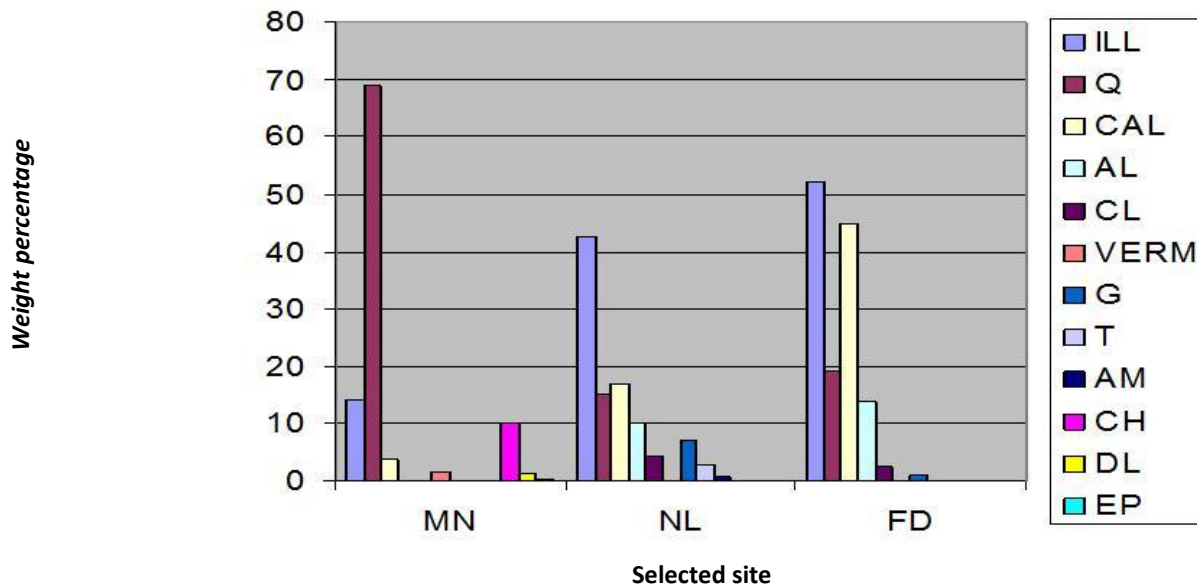
Table 7. Weight percentages of phases present in Special sample (S-2).

Phases identified	Integrated Intensity (I _i)	Weight percentage
Q	436.8	64.61
AL	BDL	BDL
ILL	320.2	28.08
CAL	60.6	5.32
CL	BDL	BDL
T	BDL	BDL
G	BDL	BDL
CH	BDL	BDL
AM	BDL	BDL
EP	BDL	BDL
DL	BDL	BDL
VERM	22.7	1.99

Table 8. pH values, suitability and acidic zone identification of soil samples.

Selected sites	pH values (suitability of the soil)	Probability of acidic zone identification
Multan	8-9 (unfavorable)	41.86% LPZ
Narowal	6 (very favorable)	8.72% HPZ
Faisalabad	7-8 (favorable)	49.42% MPZ

LPZ- low polluted zone;
 HPZ-high polluted zone;
 MPZ- medium polluted zone.



Graph 1. Comparison of weight percentage of phases present in the soil samples of Multan, Narowal and Faisalabad.

Key: In above graph, along x-axis the abbreviations of cities are: MN=Multan, NL=Narowal, FD=Faisalabad.

amount in the soil. From the plot of the comparison of the mean weight percentage of phases, it was clear that Illite was the major phase with the largest amount (53.27% and 42.62%) in the soil of Faisalabad and Narowal respectively while Quartz (SiO₂) appeared as the major phase with the largest amount (68.83%) in the soil of Multan (Graph 1). The phases responsible for the fertility of soil were Illite, Calcite, Chlinochlore, Vermite, Gypsum, Chamocite, Dolomite and Epidote due to the fact that they have primary, secondary and micronutrient elements, while Quartz, Albite and Aluminat did not play much role in the fertility of soil Shahid et al., 2008, 2012², 2012³). So values of these phases were ignored in the aggregate mean weight %age values. The aggregate mean weight %age value of phases responsible for fertility in the soil of Faisalabad was 67.23, while in the soil of Narowal it was 73.54 and in the soil of Multan it was 21.14. It was observed that the aggregate mean weight percentage value of phases responsible for fertility

in the soil of Narowal was greater than that of the soil of Multan and Faisalabad; so as far as the pH value is concerned Narowal soil was more fertile than Faisalabad and Multan. After that Faisalabad soil was more fertile than the soil of Multan. According to the XRPD analysis of soil samples, Multan soil was least fertile because it has large amount of Quartz. The soil of Narowal was best fit for irrigation purposes since it has pH values of 6, 8-9 and 7-8 respectively, while the soil of Multan and Faisalabad was not best fit for irrigation purposes (Hemming and Klas, 1998). The pH value result also supports the quantitative phase analysis result. The special sample S-1 showed that some minerals which were already present in the soil of Narowal and Faisalabad disappeared after combining the soil samples which means that there was a special phase found in the sample which not only became the cause of disappearance of minor phases but also affected the values of major phases. The appeared phases were Q,

CAL and ILL, while the disappeared phases were AL, CL, G, T and AM. In the same way, for the special sample S-2, CH, DL and EP did not appear after combining all the samples of Multan. From the analysis of both samples, it was clear that there were some minerals which disappeared with the minor phases and then appeared before making the special samples (Long and Stevens, 1996; Markewitz and Richter, 2000; Novaseqhi, 1998; Plancon and Drit, 2000; Rocha and Gomes, 1995; Skrovarov et al., 1996; Shahid, 1997). Strictly speaking, soil texture provides important information about the plasticity, rigidity, permeability, droughtiness, fertility and producibility. In this way, it becomes an essential characteristic just as color and physio-chemical composition. The texture analysis of soil samples from selected areas confirmed that soil was composed of 50.25% clay, 30.95% silt, 18.6% sand and 8% organic matter. This confirms the dominance of soil minerals over other ingredients of the soil and hence proves the universality of XRD technique (Gupta, 2009; Deshmukh, 2012).

Conclusion

- The phases present in the soil samples of Faisalabad were Illite, Quartz, Calcite, Albite, Chlinochlore and Gypsum, while those present in the soil samples of Narowal were Illite, Quartz, Calcite, Albite, Chlinochlore, Talc, Aluminate and Gypsum, and those present in the soil samples of Multan were Illite, Quartz, Calcite, Vermite, Chamosite, Dolomite and Epidote.
- Major phases in the soil of Faisalabad and Narowal were Illite, Quartz, Albite and Calcite, while major phases in the soil of Multan were Quartz, Illite and Chamosite.
- Illite was the top most major mineral in the soil of Faisalabad and Narowal while Quartz was the top most major mineral in the soil of Multan.
- The fertility of the soil samples followed the pattern: Narowal>Faisalabad>Multan on behalf of their pH values.
- Almost all the minor phases which were present in the selected individual sample of the soil disappeared in the special sample (combination of samples) of the soil and some new major phases reappeared which means that soil mixing may change the fertility of the soil.

FUTURE RECOMMENDATIONS

The soil of Multan and Faisalabad or any other place can be made better for fertility by increasing the quantity of K, N, Ca, Mg, S and P in it, so the compounds consisting of these elements should be used as supplements in the soil. However, random sample mixing may improve the quality of the soil through readjustment of the phases. Hence, the technique used by soil experts in gardens and parks is confirmed and may be used for general purposes also.

In the other relevant studies, it was suggested that

adequate drainage and leaching, crop rotation, blending of saline water with good qualities of water, use of manures and mulching and desiltation of Ojhar weir could be adopted to improve the soil fertility of the area, but nobody to the best of our knowledge has claimed soil mixation idea. The novelty of mixation technique is that the deficiency of minerals in our soil samples will be recovered by the other soil samples and both becomes at par for irrigation purpose. However, it can be achieved only if proper methodology for mineral study will be made along with water conservation strategy and be adopted by the farmers. The farmers should be educated about the efficient use of mixation for mineral deficit and mineral excess soil, as per their requirement. So these precious soil resources should be utilized for the best economical yield, which pose as an urgent need for developing countries like Pakistan. Thus, in this way, this study becomes more important than the previous studies on behalf of its simplicity and authenticity.

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