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Characterization, Beneficiation, and Potential Utilization of Ayubia Glauconite, Abbottabad, Pakistan

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Abstract

The glauconite deposits from Thub top near Ayubia, District Abbottabad (Pakistan) are characterized for their mineral contents, chemical composition, and beneficiation potential by using petrography, chemical analysis and high intensity wet magnetic separation. The petrographic analysis showed that the glauconitic sandstone is dominantly comprised of quartz, carbonate cements, and glauconite. Iron oxides has partially replaced the glauconite. The chemical analysis revealed a low K_2O content of 3.23%, below the threshold for economic extraction and utilization in potassium fertilizer (K-fertilizer) production. In addition, the sample in its natural form had low P_2O_5 and iron oxide contents. The upgradation of glauconite showed an increased concentration of Fe_2O_3 and FeO (iron oxides) from 11.36% to 17.79% and 4.32% to 4.50% respectively. However, there was an insignificant increase in the concentrations of K_2O, P_2O_5, and other oxides. Furthermore, the potential utilization of the beneficiated glauconite samples as K-fertilizer and water softener was evaluated. The results indicate the limited suitability of glauconite as a fertilizer because of its low potassium (K) contents. In its original form, the material displayed a negative water-softening property. Nevertheless, after regeneration through immersion in NaCl and NaOH solutions, a significant improvement in the water-softening capability of glauconite was observed and the treated glauconite reduced water hardness from 916 mg/L to 300 mg/L.

Keywords: *Glauconite, Beneficiation potential, Water Softener, K-Fertilizer; Petrographic Analysis; Chemical analysis*

1. Introduction

Glauconite $[(K,Na)(Fe,Al,Mg)_2(Si,Al)_4O_{10}(OH)_2]$ is a hydrous, dioctahedral, iron-rich silicate containing various amounts of magnesium, sodium, potassium, and trace elements (Majumder et al., 1995). It is generally olive green to greenish black. Glauconite-rich sediments are commonly known as greensand and have been considered to belong to the group of both mica and clay minerals (Rawlley, 1994). Glauconite can remove contaminants and salts from water as well as from landfill leachate due to its base exchange properties (Hao et al., 1987; Smith et al., 1996; Ahmadirouhani and Samiee, 2014; Franus and Bandura, 2014, Naghipour et al., 2020, Nassar et al., 2021). Furthermore, it is considered to have the potential to supply potassium to plants, and therefore, many researchers have recommended it as an alternate source of K-fertilizer in various countries (McRae, 1972; Mazumder et al., 1993; Rawlley 1994; Rao and Rao, 1999; Chandra, 2001; Amorosi et al., 2007; Karimi et al., 2012; Shekhar et al., 2020). The use of glauconite as a water Softener or as a K-fertilizer is dependent upon its inherent mineral composition, its form of existence within glauconite association with other minerals. The form in which minerals exist within glauconite can influence their availability for plant uptake or their effectiveness in water purification. Similarly, associated minerals may enhance or hinder the release of potassium from glauconite, impacting its availability for plant uptake (Levchenko et al., 2008; Karimi et al., 2012; Martemyanov et al., 2021). However, in its natural form, the fraction of glauconite in its parent rock hardly exceeds 20%, making it necessary to enhance its concentration before it can be used for the above-mentioned purpose using beneficiation methods (Choudhury et al., 2022).

Deposits of glauconite in the form of glauconitic sandstone have been reported at Thub top near Ayubia district, Abbottabad (Pakistan). These deposits belong to Lumshiwal Formation showing a thickness of about 12.80 meters and a lateral extension of about one kilometer. The formation mainly comprises of very fine to coarse-grained sandstone which is blackish brown, dark greenish grey, and mustard brown to reddish grey on a fresh surface. At places, the fresh surface shows a greenish tinge which becomes darker green on wetting. The type locality of this formation is about one kilometer north of Lumshiwal nallah (Shah, 1977). The formation is characterized by four basic lithologies including marl, shale, sandstone and hematitic sandstone. The glauconitic sandstone occurs towards the base where the formation has a gradual contact with the Chichali Formation (Munir et al., 1990). The geological map of the study area showing the sampled location is given in Fig. 1.

The availability of glauconite deposits at Thub top has prompted interest in the investigation of its possible use as a soil conditioner/fertilizer and water softening material. This research aimed to characterize the chemical and mineralogical composition of glauconitic sandstone and assess its potential for beneficiation. The mineral was subjected to beneficiation processes, and the properties of the resulting concentrate were evaluated for its suitability as a K-fertilizer and water softener.

2. Materials and Methods

A representative sample was collected from the Thub top section of Lumshiwal formation exposed near Ayubia, district Abbottabad (Pakistan).

Lumps up to 8 inches in size were picked from different parts of the deposit and the total sample weighed above 50 kg. A fresh sample of the glauconitic sandstone is fine to medium-grained quartz arenite (Fig. 2). The fresh color of the rock is generally green to dark green which is mainly due to the presence of a significant amount of glauconite.

The sample was crushed in a Denver laboratory jaw crusher with a set opening of about ¾ inches and the product was obtained in the form of pieces varying from ½ - ¼ inches in size. The jaw crusher product was divided into two portions. One part was retained for later use while the other half was passed through a disc crusher properly set to avoid over-grinding. The product of the disc crusher was subdivided through a riffle sample splitter into 16 portions which were packed, labeled, and preserved for subsequent analysis.

The mineralogical characterization of the sample was performed using a stereoscopic microscope. For this purpose, the sample was analyzed in powdered form under the microscope. Additionally, thin sections of the sample were meticulously prepared, and photomicrographs were captured to analyze grain size and texture. The chemical composition of the sample was ascertained through traditional wet chemical analysis methods employing titration equipment and a spectrophotometer.

A portion of the sample was subjected to complete sieve analysis ranging from +20 to -250 mesh. The liberation studies carried out on each fraction under the microscope revealed that the sample with a particle size ranging from -100 to +200 mesh was suitable for liberation. The beneficiation of the sample was analyzed by considering various separation processes.

The water-softening potential of the sample was performed using probe test. In this test, initially, 500 grams of material was soaked in 1 liter of water of known hardness (555 mg/L) and given 24 hours of contact time to facilitate the ion exchange process. After 24 hours, the filtered water was tested for its response by titration method. Ethylenediaminetetraacetic acid (EDTA) at a concentration of 0.1 M was used as a titrant. A 25 mL sample was added to 25 mL of distilled water with a few drops of buffer solution to maintain the pH of the sample within the range. After adding a tinge of Eirochrome Black T., the sample exhibited a distinct purple color. The sample was treated against EDTA until the endpoint was reached, which was indicated by a blue color. The hardness of the sample was measured by using Equation 1 as follows.

$$\text{Hardness (mg/L)} = \text{mL of EDTA used}/25 \text{ mL of sample} \times 1000 \qquad (1)$$

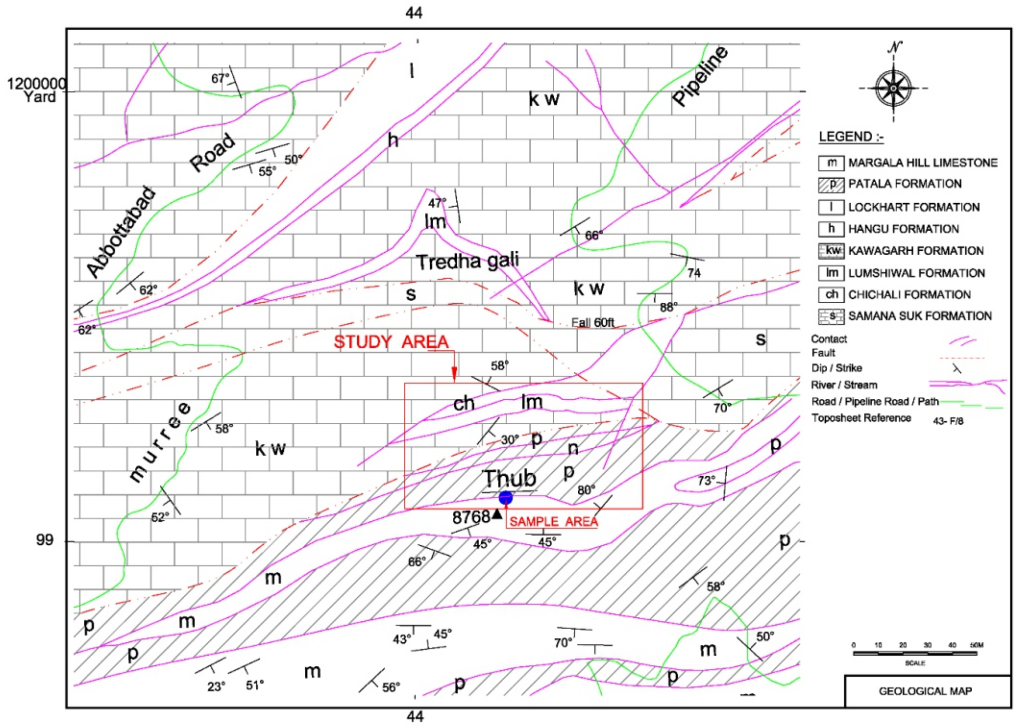

Fig. 1. Geological map of the study area showing sampled location (modified from Munir et al., 1990).

Fig. 2. Hand specimen of Glauconitic sandstone investigated in this study.

3. Results & Discussions

3.1 Physical properties

The Glauconite samples investigated in this study were predominantly fine to medium grained quartz arenite. The sample exhibited typically green to dark green color and polylobate to rounded or tabular in shape. It showed a dull or glistering luster and was opaque. Seven small pieces from the second portion of the product of the jaw crusher were taken to measure specific gravity. The measured specific gravity of a total of seven samples was found to vary from 2.2 to 2.4 with an average value of 2.27. The sample had a Mohr's scale hardness of 2. The predominantly associated minerals found in the hand specimen included quartz, glauconite, dolomite, iron oxides and clay minerals.

3.2 Petrographic characteristics

Thin sections of the sample were prepared, and their photomicrographs were studied for mineral composition, grain size, and texture. A typical photomicrographs of the sample showing appearance of various minerals is shown in Fig. 3. The glauconitic sandstone investigated in this study is petrographically fine-grained and moderately well-sorted, texturally sub-mature. It is predominantly consisting of quartz (Q) and glauconite (G) while minor minerals detected as trace to accessory concentration include sphene (S), potash feldspar (microcline, albite, and plagioclase). Carbonates occurred as the main cement while iron oxides (Feo), chert (C) and quartz (Q) were subordinate cement (Fig. 3). Where present, dolomite (D) was revealed as the main constituent of carbonate cement. It was found that glauconite had corroded and was partially replaced by iron oxides. Based on texture and mineralogy, the studied rock is classified as mature quartz arenite (Pettijohn et al., 1987). The contacts of some quartz grains were sutured, and a few quartz grains showed fractures. Veins of calcite and ferroan calcite were also observed in some thin sections. Muscovite flakes were crenulated and bent.

The modal mineralogical composition of the sample was analyzed based on thin sections, and the corresponding results are depicted in Fig. 4. A comprehensive presentation of these findings is provided in Table 1.

The petrographic thin sections reveal the prominent mineral compositions in different plates:

- Plate (a): Glauconite (35%), Quartz (20%), Dolomite and Sphene (13%)

- Plate (b): Microcline (26%), Sphene (23%), Glauconite (13%)

- Plate ©: Glauconite (25%), Quartz (18%), Dolomite (15%), Iron Oxide (14%), Sphene and Microcline (10%)

- Plate (d): Quartz (64%), Sphene (15%), Plagioclase (9%), Glauconite (3%)

- Plate (e): Quartz (62%), Plagioclase (14%), Sphene (12%), Microcline (5%), Glauconite (2.5%)

- Plate (f): Quartz (63%), Sphene (14%), Plagioclase (12%), Glauconite (2%)

- Plate (g): Quartz (70%), Plagioclase (13%), Dolomite (5%), Glauconite (3.5%)

- Plate (h): Quartz (72%), Plagioclase (12%), Microcline (4%), Glauconite (4%)

- Plate (I): Quartz (62%), Plagioclase (12%), Sphene (7%), Glauconite, Iron Oxide, and Microcline (5%)

One noticeable trend is the prevalence of quartz, which is the dominant mineral in most samples, reaching as high as 72%. Glauconite content varies from 2% to 35%, and iron oxide content ranges from 1% to 14%. Additionally, significant quantities of other minerals, including Sphene, Plagioclase, Microcline, and chert, are also present.

3.3 Chemical characteristics

The complete chemical analysis results of the collected glauconitic sandstone sample are presented in Table 2. The data indicates that the glauconite from Thub top, Ayubia, Abbottabad exhibits a significantly low K_2O content, measuring only 3.23%. This concentration falls below the threshold for economic extraction and utilization in K-fertilizer production using the current technology. The literature suggests that the minimum P_2O_5 and K_2O contents for qualifying as fertilizer should be 10 % and 15% respectively (McLaurin and Reeves, 2009). Additionally, the glauconite sample contains 11.36% Fe_2O_3 and 4.32% FeO, which, when considered together, do not meet the criteria for categorizing it as a potential low-grade iron ore deposit in its natural form (du Plessis et al., 1997). Overall, the chemical analysis results demonstrate that the studied sample lacks the necessary concentrations of potassium and iron to be economically viable for manufacturing K-fertilizer or to be classified as a potential low-grade iron ore deposit.

3.4 Beneficiation

Grain mounted glass slides were studied under microscope. The scrutinizing study revealed that glauconite (green color sand) was not liberated from quartz adequately up to 80 mesh. However, when it is further grinded to pass 100 mesh, the liberation appears to improve. It was fair enough (~60%) at -150 mesh and looked adequate (~80%) at -170 mesh to +200 mesh. At 250 mesh, the glauconite sample was too fine to be used for concentration. Therefore, depending on the microscopic study, the material between –100 mesh to +200 mesh was considered suitable for the selection of the separation process. However, considering the specific gravity of glauconite (2.27 g/cc) and quartz (2.65 g/cc), the exploitable difference in specific gravity between glauconite and quartz was found to be insignificant (0.38). Consequently, gravity separation was considered unsuitable. Glauconite, owing to the presence of iron, is typically upgraded through magnetic separation. However, in this study, dry magnetic separation was not viable due to sufficient liberation being limited to below 150 mesh. An attempt was made to utilize a high-intensity induced rolls magnetic separator; however, no concentrate was observed. This lack of concentration may be attributed to the possibility that extremely fine particles adhered to the original glauconite particles, thereby concealing their distinctive properties. Consequently, a Carpco machine, Model number WHIMS 3×4L, known for its high-intensity wet magnetic separation capabilities, was utilized. The WHIMS 3×4L, unit is designed for operating on 120 or 220 VAC,50/60 Hz and single phase. This instrument yielded a concentrate that appeared satisfactory because the weight recovery of the concentrate reached approximately 65%. The Carpco machine operated at the highest field level of 4 Amp and the magnetics and non-magnetics were both recycled two times each to obtain the final concentrate.

The oxide analysis of the concentrate and feed are presented in Table 3. The results indicated that the iron oxide content was increased in the concentrate from 11.36 to 17.79 % in the case of Fe_2O_3 and from 4.32 to 4.50 % in the case of FeO giving a combined iron oxide concentration of 22.29%. Hence, the concentrate meets the criteria for categorizing it as a potential low-grade iron ore deposit (20% to 47%) (du Plessis et al., 1997). The fertilizer-making oxides such as K_2O and P_2O_5 did not show any substantial improvement, rather the content of P_2O_5 was decreased in the concentrate. Moreover, aluminum oxide also showed a little improvement in its grade. Very little effect on other oxides was observed.

3.5 Evaluation of glauconite as a conditioner/Fertilizer

The soil conditioning property of the material depends upon potash and phosphate content. The P_2O_5 and K_2O content of the studied rock samples were 0.31 and 3.23 respectively as shown in Table 3. These values are far below the minimum contents for qualifying as fertilizer ($K_2O \geq 10$ %) and ($P_2O_5 \geq 15\%$), this shows that the studied rock is a poor-quality mineral deposit for utilization as fertilizer or water softener. Unfortunately, both the natural material and its concentrate fall short of the desired specifications. Further, the sample was tested for its soluble content of K_2O and P_2O_5. The water-soluble contents of both oxides were found to be almost zero. However, a digestion test by perchloric acid indicated 3.26 % K_2O and 0.025 % P_2O_5. The above laboratory results disqualified the investigated indigenous material for any use as a fertilizer or soil conditioner. No further test work in this connection was considered.

3.6 Evaluation of glauconite as a water-softening material

The probe test was conducted to determine the response of glauconite toward water softening. It was found that the hardness was increased from 555 mg/L to 705 mg/L. In the second phase, the time of contact was further increased to give a maximum time (chance) for the ion exchange process, but unfortunately, the hardness was further increased to 750 mg/L. The increasing hardness of water showed that further hardness was coming from the material itself. In the third phase, 500 grams of material was soaked in 1 liter of distilled water and waited for 24 hours. After 24 hours, the filtered water was tested for its hardness. It was found that the hardness increased from zero to 88 mg/L. The tests were performed with both original glauconite sample and concentrate, the results remained the same. This indicated that some water-soluble ions possibly calcium (Ca^{2+}) and magnesium (Mg^{2+}) were released from the sample. In the fourth phase, the test samples were soaked in saturated NaCl solution for 24 hours, washed with distilled water, and filtered. A hard sample (916 mg/L) was treated, and it showed a sufficient softening effect, as the hardness reduced from 916 mg/L to 300 mg/L. In the fifth phase, the sample was treated with a saturated solution of NaOH for 1 hour and after washing, its water-softening property was tested. The hardness was found to reduce from 916 mg/L to 400 mg/L. Finally, the sample was soaked for 24 hours with NaOH solution and then the softening test was performed after washing it with distilled water. The hardness of the sample was found to decrease significantly from 916 mg/L to 300 mg/L.

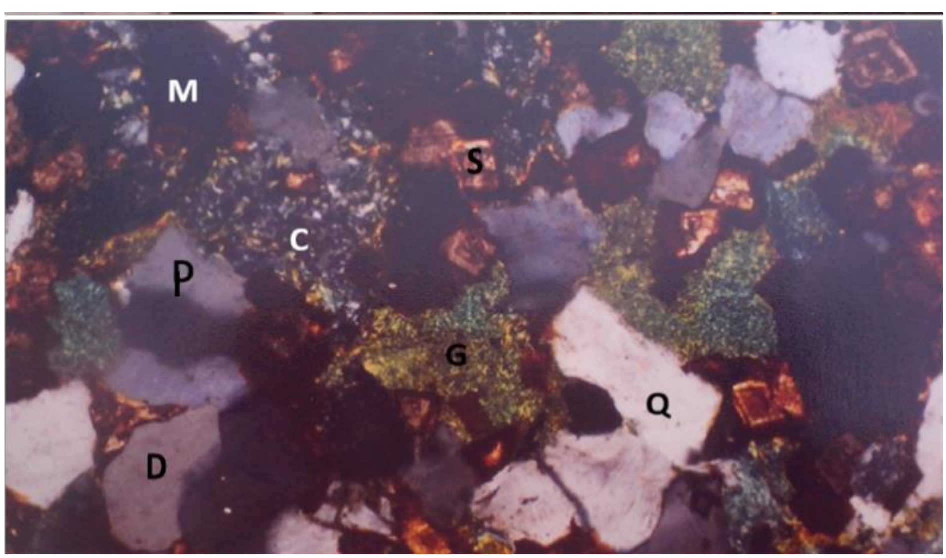

Fig. 3. Microphotograph of the studied sample showing texture and minerology of various minerals: Glauconite (G), Quartz (Q), Dolomite (D), Sphene (S), Microcline (M), Chert (C), Plagioclase (P).

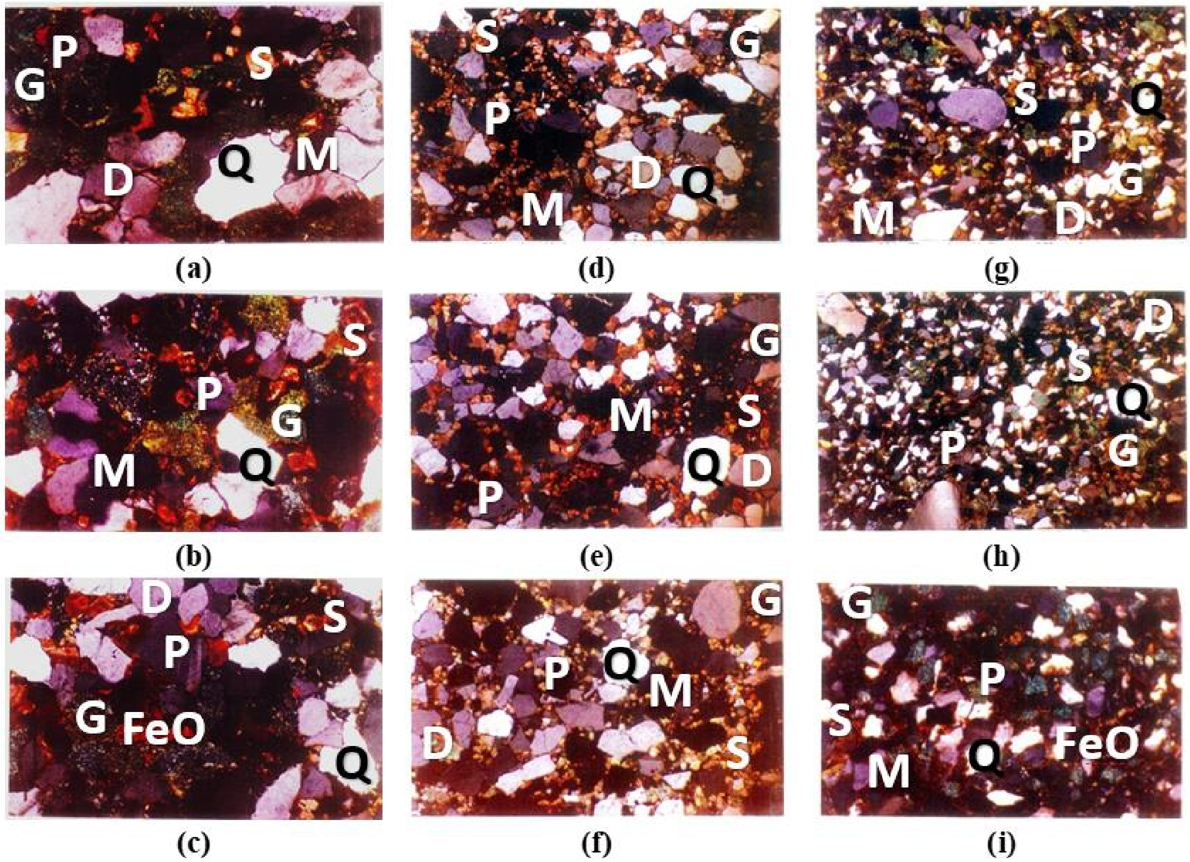

Fig. 4. Microphotographs of the thin sections of the studied glauconitic sandstone sample

Table 1. Modal mineralogical composition of the studied glauconitic sandstone samples.

Mineral	Plate (a)	Plate (b)	Plate (c)	Plate (d)	Plate (e)	Plate (f)	Plate (g)	Plate (h)	Plate (i)
Quartz (%)	20	12	18	64	62	63	70	72	62
Glauconite (%)	35	13	25	03	2.5	02	3.5	04	05
Dolomite (%)	13	09	15	02	2.5	04	05	03	03
Sephene (%)	13	23	10	15	12	14	03	03	07
Plagioclase (%)	08	08	07	09	14	12	13	12	12
Iron Oxide (%)	02	04	14	2.5	02	02	2.5	01	05
Microcline (%)	09	26	10	3.5	05	03	2.5	04	05
Chert (%)	-	05	01	01	-	-	0.5	01	01

Table 2. Chemical composition of glauconitic sandstone sample

Sr. No.	Major Oxides	Percentage
1	SiO_2	63.12
2	TiO_2	0.50
3	Al_2O_3	11.71
4	Fe_2O_3	11.36
5	FeO	4.32
6	MnO	0.02
7	CaO	0.10
8	MgO	0.93
9	Na_2O	1.21
10	K_2O	3.23
11	P_2O_5	0.31
12	SO_3	1.26
13	L.O.I*.	1.93

*L.O.I. Loss on Ignition

Table 3. Compositional analysis of feed and concentrate

Sr. No.	Major Oxides	Feed	Concentrate
1	SiO_2	63.12	55.79
2	TiO_2	0.50	0.52
3	Al_2O_3	11.71	13.13
4	Fe_2O_3	11.36	17.79
5	FeO	4.32	4.50
6	MnO	0.02	0.01
7	CaO	0.10	0.10
8	MgO	0.93	1.09
9	Na_2O	1.21	1.23
10	K_2O	3.23	3.46
11	P_2O_5	0.31	0.27
12	SO_3	1.26	0.95
13	L.O.I.*.	1.93	1.16

*L.O.I. Loss on Ignition

4. Conclusions

The mineralogical characterization showed the presence of glauconite (upto 35%) associated with quartz, dolomite, plagioclase, microcline, iron oxide, sphene, and chert. The results of the chemical analysis revealed that potassium oxide and phosphorous oxide were found to be less than the minimum value required for its usage as a soil conditioner or fertilizer. The beneficiation of the mineral resulted in a 57% increase in Fe_2O_3 content and 4% increase in the FeO content in the concentrate. The fertilizer-making oxides such as K_2O and P_2O_5 and other oxides did not show any substantial improvement. The initial prob test results indicated that the glauconite itself contributed to water hardness as demonstrated by an increase from555 mg/L to 705 mg/L. However, it was observed that prior treatment of the glauconite sample with saturated NaCl and, particularly, NaOH substantially enhanced its water-softening properties. This enhancement was evidenced by a significant reduction in water hardness, lowering it from an initial level of 916 mg/L to 300 mg/L after treatment.

Authors' Contribution

Shahab Saqib was responsible for proposing the core concept and conducting the experimental work. Muhammad Shehzad and Shahab Saqib created the initial draft. Shahab Saqib and Hafiz Muhammad Awais Rashid did provision of relevant literature, reviewed and proofread the manuscript for the final draft. Muhammad Farooq Ahmed supported in collecting field data and obtaining samples, Muhammad Mansoor Iqbal provided essential support for the experimental efforts.

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Conflict of Interest

The authors declare that they have no potential conflict of interest regarding this research work.

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