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Investigation of microwave roasting for potash extraction from nepheline syenite

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ABSTRACT

Nepheline syenite is an igneous rock, which contains around 5.4% of potassium oxide (K_2O), but finely dispersed and, locked in the host matrix. The characterization studies reveal the presence of orthoclase, nepheline and biotite as the major potassium bearing mineral phases. The present study aims at the extraction of potash values through microwave heating for its possible application as fertilizer. The microwave assisted roasting studies of nepheline syenite have been carried out and optimized using a statistical approach based on Taguchi design. It is possible to extract around 88% K_2O values at 900 W microwave power and 6 min of roasting time using 20% charcoal and 50% calcium chloride. Charcoal is used as a microwave absorbing material that raises its temperature in a much lesser time, while calcium chloride for the favorable formation of KCl. The transformation of different mineral phases of nepheline syenite is characterized using XRD, and SEM showing the formation of a soluble potassium bearing phase (Sylvite) along with calcium aluminum silicate.

1. Introduction

The rising demand of potash for the growth of plants, human and animal life is inevitable [1]. Unfortunately, there are no potash resources such as sylvite, carnallite or polyhalite in a vast country like India. Therefore, it imports all its potash demand (about 3.5 million tons per annum) from different countries. India possesses abundant mineral sources like glauconitic sand, feldspar, muscovite, and nepheline syenite containing 3–14% K_2O. However, no systematic effort has been made to recover the potash values from any of these available resources. The recovery of potassium from the silicate rocks has received little attention probably due to the slow release of potassium values, the availability of appropriate economic processes, and their know-how. Some efforts have been made to recover potassium values from feldspar and glauconitic sandstone by acid leaching, and calcium chloride roasting followed by water leaching [2–5]. However, to the best of our knowledge, no commercial extraction of potassium values from nepheline syenite has been reported so far.

Nepheline syenite is a feldspathic and plutonic igneous rock. It is primarily made up of nepheline $(Na,K)AlSiO_4$, feldspar, albite ($NaAlSi_3O_8$), and orthoclase ($KAlSi_3O_8$) [6]. It is used as a source for Al_2O_3, Na_2O and K_2O for glass and ceramics industry. In ceramic industries, nepheline syenite is used as an active agent for the formation of a glassy phase in the ceramic body and provides the physical strength to the final products. The K_2O content in nepheline syenite varies from 3% to 14%. Most of the investigations on nepheline syenite are concentrated on the removal of iron values by magnetic separation and flotation techniques for its use in glass and ceramics production [6,7].

Microwave energy based on non-ionizing electromagnetic radiation with frequencies in the range of 0.3–300 GHz is an alternative source of heating. The microwave frequency in the range of 2.45 GHz is widely used in different industries. Microwaves generate molecular motion by migration of ionic species and rotation of dipolar species. Microwave heating is material specific, which depends on the ratio of the dielectric loss and the dielectric constant termed as dissipation factor. The major advantage in microwave treatment is the non-contact energy transfer compared to the direct heat transfer in conventional heating. The rapid heating generated by microwaves can be transmitted, absorbed or reflected depending on the nature of the material [8,9]. Some of the major mineral engineering applications of microwave energies include the treatment of ores with complex mineralogy in order to enhance the liberation, microwave assisted grinding, and carbothermic reduction of ores. The process of heating followed by sudden quenching causes the micro cracks, which decreases the lattice strength of the ore and thereby reduces the grinding cost. One major disadvantage of conventional way of heating adopted in

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industries is that it requires a significant amount of heat input; also the energy balance is unfavorable due to the loss of energy [10]. On the other hand, microwave heating offers advantages over the conventional heating as it selectively heats the responsive mineral in the ore and no heat loss occurs due to bulk heating. In this context, it has been reported that the ore mineralogy has a significant impact due to the selective heating of allied phases and thus high temperature heating can be avoided [11–14]. The effect of pre-treatment of microwave on grindability of different ores such as coal, oolitic iron ore, and tin has been reported beneficial due to the development of micro fractures along the boundaries, and reduced bond work index [11,12,15,16]. The efficiency of microwave pre-treatment is dependent on the microwave power and time of exposure.

Besides grinding, microwave heating also finds some broad application in carbothermic reduction processes of metal oxides. It offers faster heating rate, uniform heat distribution, and preferential reduction that start from the core to the surface as compared to the reverse direction in conventional heating. Therefore, microwave methods are reported as an efficient alternative for the reduction of metal oxides [17–19]. The use of microwave pre-treatment prior to leaching has also been investigated by many researchers [20,21].

Considering the above mentioned applications of microwave energy, an attempt has been made to extract potassium recovery from nepheline syenite by using microwave assisted heating technique. The purpose of using microwave in place of the conventional roasting is because of its ability to offer faster kinetics through rapid heating. To the best of our knowledge, this methodology has not been applied to optimize the potash extraction from nepheline syenite till date. Recently, we have reported the extraction of potash values from nepheline syenite by $CaCl_2$ roasting in a conventional furnace at ~900 °C followed by water leaching [22]. Although it was possible to recover ~99.6% of K_2O values, it requires higher temperature and time. Hence, in the present study, an attempt has been made to use microwave energy with an objective to reduce the energy consumption in terms of time and temperature.

2. Materials and methods

2.1. Sample pre-treatment and characterization

Nepheline syenite sample was collected from one of the mines in Odisha, India. The size reduction of the sample was carried out by using stage crushing with laboratory jaw crusher and roll crusher. The laboratory jaw crusher was supplied by Eastman Crushers Company (P) Ltd, Kolkata India, where as the roll crusher from Rajco Science and Engineering product, New Delhi, India was used in the crushing studies. The size analysis of the crushed products was carried out by standard sieves down to 100 μm. The 80% passing size of the jaw crushed and roll crushed products were found to be 35 and 1 mm respectively. The sample was further ground in a laboratory ball mill of 300 × 300 mm size for the different experimental studies. The grinding was carried out in batch with different intervals of grinding time. At the end of the each grinding time, the product was taken out and subjected to size analysis using standard sieves. The size analysis of the ball mill ground products were carried out by using the sieves from 500 μm down to 25 μm. The 80% passing size of the ball mill ground product after 10 min of grinding time was found to be 150 μm.

The iron, alumina and silica content of the ore were determined by wet chemical methods, whereas the elemental analysis of potassium, sodium and calcium were done by a Systronic flame photometer (model 128μc). The finely ground dried sample (~1 g) was used for the chemical analysis. It was digested in a Teflon beaker using hydrofluoric acid (5 ml) and nitric acid (2.5 ml). The sample was dried well in order to remove all the acid present in the treated sample. Then, it was redissolved using diluted hydrochloric acid and boiled for 5 min, and filtered to a volumetric flask. The solution was diluted for the analysis purpose. The K, Na and Ca contents of the sample were analyzed using the flame photometer, and the results were confirmed by ICP–OES using the certified standards. In both the cases, the results were found to be same. The analysis of the sample was also checked in other certified laboratories. The other constituents such as silica, alumina and iron oxide, were analyzed after fusing the sample with sodium carbonate. The Al and Fe content of the sample were determined by titrimetric method while the silica was determined by gravimetric methods. The loss on ignition (LOI) of the sample was determined after igniting a known weight of sample in a muffle furnace at 950 °C as per Indian standard specifications. The major and minor elemental analysis of the sample is shown in Table 1. The sample contains 5.4% K_2O, 56.8% SiO_2, 14.6% Al_2O_3 and 11.1% Na_2O.

The morphological appearance of nepheline syenite sample was investigated by a scanning electron microscope (SEM) and the mineralogical phases were determined by X-ray diffraction (XRD) studies. The JEOL JSM 6510 scanning electron microscope was used to analyze the surface morphology of nepheline syenite and the microwave roasted product at 1500 times magnification. After surface cleaning with absolute ethyl alcohol, the nepheline syenite samples were dried in air. The samples were sputter-coated with a layer of gold prior to the SEM studies. The XRD studies of the samples were carried out using the model, Rigaku Ultima IV, X-ray diffractometer at Co Kα radiation. The diffractograms were recorded from 10° to 80° with a scan speed of 30 s/step to identify different mineral phases present in the sample.

Microscopic studies along with XRD data on the nepheline syenite sample indicated the presence of albite, anorthite, microcline, nepheline and biotite as the major phases. The iron content in the sample, as indicated in Table 1, is due to hematite and biotite. The sodium content in the sample is primarily due to albite. The microcline and nepheline minerals contributed significantly to the potash values in the sample.

2.2. Microwave treatment method

The experiments were carried out using a 2650 MHz domestic microwave (LG make, model MC 2302FUPG) oven of 30 l capacity having maximum 900 W of power output. The calcium chloride di-hydrate (98% purity) was used in this investigation for the transformation of potassium into a soluble chloride. The activated charcoal (AC) used in the experiment was of 96% purity. It consisted 3% acid soluble matter, 0.02% water soluble matter, 0.2% Fe, 0.2% chlorides and 0.2% sulfates. For each designed experiment, premeasured amounts of feed sample, charcoal and calcium chloride were rigorously mixed. All the experiments were carried out using

Table 1
Chemical constituents of nepheline syenite.

Components	Percent (%)
Fe_2O_3	2.82
Na_2O	11.10
K_2O	5.40
SiO_2	56.80
Al_2O_3	14.60
CaO	3.84
LOI	1.02