Chemsearch Journal 2(1): 5 - 7
Publication of Chemical Society of Nigeria, Kano Chapter

THE EFFICIENCY OF MIXED ACIDS AND SODIUM PEROXIDE IN THE DECOMPOSITION OF RAW KAOLIN

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ABSTRACT

The study compared decomposition efficiency of mixed acids and sodium peroxide on raw kaolin sample. The sample was decomposed using the two methods and its chemical composition determined. Standard concentrations of elemental chemical composition obtained from the decomposition were prepared and used to spike the raw kaolin sample. The spiked kaolin sample was decomposed using the two methods. Percentage recovery of the elemental oxides show that mixed acids have higher efficiency for MgO, Na_2O and K_2O while Na_2O_2 favour FeO, CaO, Al_2O_3, TiO_2 and SiO_2.

Keywords: Kaolin, Spiking, Decomposition, Efficiency.

INTRODUCTION

Kaolin is a (2-5μm) clay material that develops plasticity upon mixing with water (Bruce and O'Hare 1992). It is predominated by the kaolinite mineral with other minerals occurring in smaller percentages (Cornelius and Klenin, 1977). Studies of Jos, Kankara and Oshiele reported percent kaolinite as (89.9, 92.5, 90.7%), muscovite (5.6, 0.75, 0.95%) and paragonite (0.0, 0.28, 0.24%) respectively (Irabor, 2002).

Processed kaolin has applications in pottery products, paper, ink, plastics, insecticides, textile and pharmaceutical (Murray, 1963). The Federal Government of Nigeria has identified 111.6 million metric tonnes of kaolin deposits in 12 states of the federation (Guardian Newspaper, 2005). Inspite of its abundance and applications, the lack of data on available raw materials has made it difficult for investors to show interest in this manufacturing sector (Irabor, 2002).

Kaolinite being a silicate mineral, the aim of its analysis was to account for total composition of the sample so that when each elemental determination was expressed as the percentage of its oxide present (e.g. Si, Al as SiO_2, Al_2O_3), the summation of all elements totalled close to 100%. To achieve this, the following determinations were carried out, SiO_2, Al_2O_3, FeO, TiO_2, MgO, CaO, K_2O and Na_2O (Newman, 1987). Such samples must be dried and ground to a fine powder before decomposition.

Sample digestion (decomposition) plays a central role in almost all analytical processes, but is not often recognised as an important step in analytical chemistry, with primary attention being directed to the determination step. This sense of priorities is reflected all too conspicuously in the equipment and investment planning of many analytical laboratories. However, a welcome trend in recent years points towards fuller recognition of the true importance of sample digestion in the quest for high quality analytical results and valid conclusions (Matusiewicz online) This study aims at determining the most efficient decomposition method for kaolin sample considering their inadequacies in some respects. Knowledge of the accurate elemental composition determines the industrial application of kaolin sample.

The two most common methods employed in dissolving samples are treatments with hydrochloric, hydrofluoric, nitric, sulphuric, or perfluoric acids (or various combinations thereof) and fusion with an acidic or basic flux followed by treatment with water or an acid. The use of single acid for decomposing a substance is not promising as each acid is limited to some extent e.g. hot, concentrated nitric acid will dissolve all common metals with the exception of aluminium and chromium, which are passive to the reagent as a result of surface oxide formation. Hence, acids in combination are preferred for dissolution of certain inorganic matrices. Wet digestion procedures using acid mixtures can be divided into four types:

a. Total decomposition, usually with hydrofluoric acid and another mineral acid.
b. Strong attacks, for routine analysis but leaving a residue of certain minerals, particularly silicates, carried out with various mixtures of sulphuric, nitric, and perchloric acids.
c. Moderate attacks, using weaker acid mixtures.
d. Partial digestions (acid leaching).

For geochemical samples containing silicates, the matrix is decomposed by heating with hydrofluoric acid in combination with either nitric or perchloric acid, each of which has a higher boiling point than hydrofluoric acid. The presence of the second acid with a higher boiling point ensures that, once the hydrofluoric acid has been boiled off and the dry sample redissolved, sparingly soluble metal fluorides are converted to salts that are more soluble (Twyman, 2005).

Kaolinites are classified as resistant mineral which hardly dissolve by acid attack. It is observed that only HF dissolves all materials. However, HF alone is not used because salts of K, Ca etc have low solubility in this acid and the resultant solution may contain other elements in an uncertain oxidation states (eg Fe). Therefore, HF mixed with aqua regia as Cl- ions reduce interference effects for AAS analysis (Potts, 1992).

Some materials are particularly resistant to acid digestion, e.g., certain rocks, mineral oxides, phosphates, and some iron alloys. For these samples, high-temperature fusion with an acidic basic or flux such as lithium metaborate (LiBO3) in the molten state can be used to render such materials soluble in water or dilute acid. Fusion decompositions are the most rigorous methods available and all silicate materials, including refractory substances like zircon and cassiterite, can be dissolved completely when fused with an appropriate flux. However, there are several disadvantages to this method including the introduction of additional salts into the final solution (Twyman, 2005).

EXPERIMENTAL

-Mixed acids decomposition: 0.1g of raw kaolin sample was weighed into 120cm^3 screw-capped polypropylene bottle and wetted with 0.2cm^3 aqua regia followed by addition of 5cm^3 40% HF. The sample was heated on a steam bath for 1:30min.when a clear solution was obtained and 50cm^3 of saturated boric acid was added. The solution was diluted to 100cm^3 and portions with their calibration standards were analysed for the oxides (Potts, 1992).

-Na_2O_2 decomposition: another 0.1g of sample was weighed into a platinum crucible and 0.3 g sodium peroxide powder was added. The two were mixed intimately with a glass rod and the charge in the crucible was covered with 0.3g thin layer of the peroxide powder. The crucible was then placed in the muffle furnace and the temperature gradually increased to 480 ± 10°C and left for 20min. to sintered cake. The cake was extracted in cold water and 60cm^3 5M HCl was added to clear the solution. The solution was diluted to 1000cm^3 and portions with their calibration standards were analysed (Jeffrey, 1975 and Van loon, 1980).

Spiking: Separate 0.1g of the sample was spiked with the standard concentration of the analytes. The spiked sample was decomposed using methods (Potts, 1992) and (Jeffrey, 1975 and Van loon, 1980), then analysed for CaO, FeO, MgO with AAS, Na_2O, K_2O by flame photometry and TiO_2, Al_2O_3, SiO_2 using UV-spectrometry (Vogel, 1961).

RESULTS AND DISCUSSION

The percent efficiency of the decomposition methods for every analyte is as shown in Table 1

Table 1: Percent Efficiency of the Decomposition Methods

Analyte	**Decomposition Efficiency (%)**	
	Na_2O_2	Mixed acids
1 FeO	93.41	85.81
2 MgO	46.68	71.18
3 CaO	99.52	86.30
4 Na_2O	88.00	96.0
5 K_2O	65.28	71.23
6 Al_2O_3	79.75	NIL
7 SiO_2	78.79	NIL
8 TiO_2	68.03	NIL

The order of decreasing efficiency for Na_2O_2 showed $CaO<FeO<Na_2O<Al_2O_3<SiO_2<TiO_2<K_2O<MgO$ while for mixed acid it was $Na_2O<CaO<FeO<K_2O<MgO<Al_2O_3=SiO_2=TiO_2$. It can be deduced that the easily dissolved components in both decomposition methods are CaO, FeO and Na_2O though with varying recovery. Their easy dissolution could be attributed to the nature of their oxides as they could all be in an exchange sites and therefore become easily dissolved by both digests(Newman 1987).

The Al_2O_3 and SiO_2 are the main framework structure of the octahedral and tetrahedral layers, they are expected to be resistant to the digest attack and therefore showed low recovery in Na_20_2 with a complete failure in mixed acids. Similarly, TiO_2 is expected to have undergone isomorphous substitution with Al_2O_3 and SiO_2 and behave as such (Newman 1987).

K_2O and MgO with least recovery even in Na_2O_2 are expected to be in the structure of impurity minerals such as micas, which are also refractory like kaolinite (Konta, 1975). The study reveals that none of the methods surpassed the other in all the elements. Also, it is observed that Na_2O_2 is most suitable for FeO, CaO, Al_2O_3, SiO_2 and TiO_2 while mixed acids favour MgO, Na_2O and K_2O. It is recommended that a combination of the two methods be used in decomposing kaolin sample for compositional analysis.

CONCLUSION

Kaolin was found to be decomposed by both Na_2O_2 and mixed acids. Spiking experiment shows different efficiency in decomposing the various elemental components of kaolin sample. It is recommended that the two decomposition methods be simultaneously used in the analysis of a kaolin sample.

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