The Extraction of Alumina from Kaolin

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Abstract- *Alumina has wide industrial and technological applications that can be extracted from different locations, different methods and materials. Kaolin from Iraq Alduikhla astrologer has used as a raw material source for alumina production. The alumina concentration in the Iraqi kaolin is more than 34% which considered as a good replacement for bauxite rack. Crushing andground is the first process to reduce the kaolin particle size to the micron level which increases the surface area of kaolin.*
The kaolin is heat treated at different temperatures (600, 650, 700 and 750) ℃ for 2 hours to remove some of ithe mpurity like organic materials and crystal water before acid treatment. A different concentration of hydrochloric acid (pH) (0.45, 0.5, 0.55, 0.6 and 0.65) has used for extraction of alumina from kaolin. The reaction between the hydrochloric acid and kaolin has studied at different temperature (30, 60 and 90) ℃. The extraction of alumina has decreased with increase t inhe reaction temperature. Finally t,,he alumina extraction by this method has characterized using XRD and XRF to investigation the crystal structure and the amount of impurities presented there. The final extracted alumina h isaving cubic crystal structure (γ alumina) with purity above 95%.

Keyword- *kaolin; extraction; Alumina.*

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1. Introduction

Alumina has wide industrial and technological applications. It presented in different crystal structures like δ, α, γ, η, κ, χ and θ [1]. The alumina has phase transformation from γ to α at a temperature range (1190-1240) °C depending on heating rate. Low heating rate leads to reduce in the phase transformation temperature. The phase transformation temperature shows 1190°C at 5 °C/min heating rate while its 1240°C at 20 °C/min heating rate [2].
The bauxite has been widely used to produce alumina for industrial application via using the Bayer process. The extraction of alumina from Bauxite by Bayer process required a high grade of bauxite ore (silica content less than 7%) because it considers the uneconomical process for production. These ores are limited abundant in few countries while nonbauxitic ores are widely spread in many countries [3-5]. The commercial process has been used for the production of alumina from low grade aluminum ore by acid treatment. Clays (kaolin) contain about 30-40% of alumina. Kaolinite is one of most interesting ore due to high alumina content, which makes it a good replacement for bauxite ore [6].
Kaolin that used in this study is from Iraq Alduikhla astrologer which contains about 34% alumina. The Iraqi kaolin has used as based materials in many application like fire bricks (refractories) and as composite materials in manufacturing nickel absorption filter [10,11]. Extraction of alumina from kaolin by leaching with acids or basis has been studied in many types of research [4-9].
Before starting the chemical treatment, the local kaolin has been calcined at different temperatures (600, 650, 700 and 750) ℃ for 2 hours. There are two advantages of using the heat treatment, the first advantage is removing the impurities like organic materials, water and chlorine gas. The second advantage is activation of alumina layer with in the kaolin structure to accelerate the leaching with acid in chemical treatment [3, 7]. In this research the γ alumina has been synthesis, its structure has been transformed to α alumina above 1200°C which is irreversible transformation. This type of alumina has wide industrial and technological applications such as catalyst for automotive, oil refinery (petroleum industry), coating materials for abrasive and thermal barrier [6]. The aim of this study is to extract the alumina from low grade materials like kaolin and purification to the high purity grade alumina above 95%.

2. Experimental work

I. The materials

Kaolin is clay mineral that has chemical composition $Al_2Si_2O_5(OH)_4$ and layered crystal structure of aluminum silicate with a hydrogen bond between the layer which gives greasy

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characteristic when it mixed with a polar solvent like water [1].

In this research, the kaolin was used which is brought from Alduikhla astrologer Iraq as based material. The chemical analysis of Kaolin is given in Table 1 show Al_2O_3=34.21% and silica=48.5% which consider the major compound, the other oxides are presented in limited amount and finally the loss on ignition (L.O.T) having 13.44 weight concentration. Mineral composition of kaolinite figure (1) consists mainly of kaolin mineral and non-clay minerals quartz. The X-ray diffraction; technique type (SHIMADZU) model (7000 maxima), Japan made has tested in the Iraq geological survey company. Kaolin has been scanned by XRD using Cu Kα as X-ray excitation medium for generated X-Ray with scanning rate (10 deg /min) from 5°-45° at 40KV applied power. The X-ray Charts of Kaolin demonstrated in Figure 1.

Table 1: chemical composition of kaolin.

Compound	Concentration (wt. %)
SiO_2	48.5
Al_2O_3	34.21
Fe_2O_3	1.33
K_2O	0.14
Na_2O	0.46
MgO	0.23
CaO	1.3
ZnO	0.2
P_2O_5	0.01
Cl_2	0.08
SO_3	0.07
L.O.I	13.44

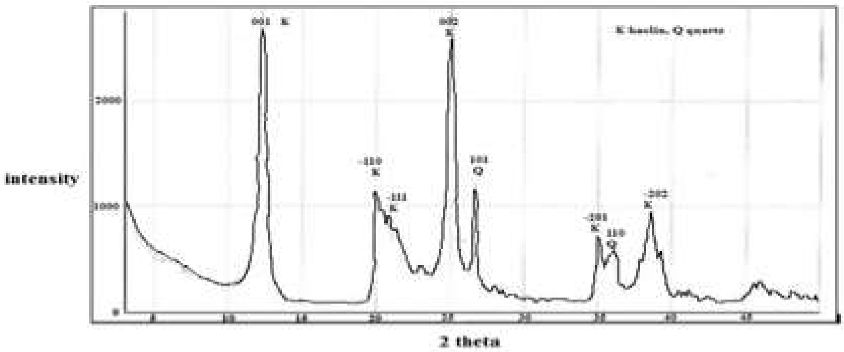

Figure 1: XRD for clay mineral kaolin and non-clay mineral quartz.

II. Crushing and Grinding

This process will contain three steps, starting with crushing the kaolin rock with grain size (10-20 inch) by using jaw crusher for a few minutes to reduce the size of kaolin rock to 1-2 inch then grinding by using porcelain ball mill for 1 hour. Table 2 shows that the particle size distribution occurred by the standard sieves for 30 minutes. Table 2 shows that the particle size distribution is in range 125µm to 38µm. All these sizes have used in this research.

Table 2: Kaolin particle size analysis

No.	Sieve size (mm)	Weight percent
1.	>0.125	13.88%
2.	0.125	29.36 %
3.	0.105	29.47%
4.	0.088	18.88%
5.	0.075	2.68%
6.	0.063	1.68%
7.	0.053	2.58%
8.	0.038	1.43%

III. Calcination of kaolin

The kaolin powders have calcined at different temperature (600, 650, 700 and 750) °C for 2hour. The calcination process is important to step for extraction of alumina because it has removed some of the impurities from kaolin and activated the alumina layer with in kaolin to enhance the reaction with acid [3]. The kaolin consists of alumina, silica, few limited oxides and loss on ignition (L.O.I) shown in Table 1. The third major constituent is (L.O.I) which consists of organic materials, humidity and crystal water. The organic materials can be removed at a temperature range between (250-400) °C while the crystal water cannot remove until 575°C was reached [12]. Figure 2 shows the crystal structure of kaolin (silica, alumina and hydroxide group (crystal water)). The calcination treatment has a great effect on the hydroxide group are present at the surface of the alumina layer, two hydroxide group is combined to form water molecular (crystal water) and oxide at the alumina layer which make the alumina layer more active to reaction with acid as explain in [3].

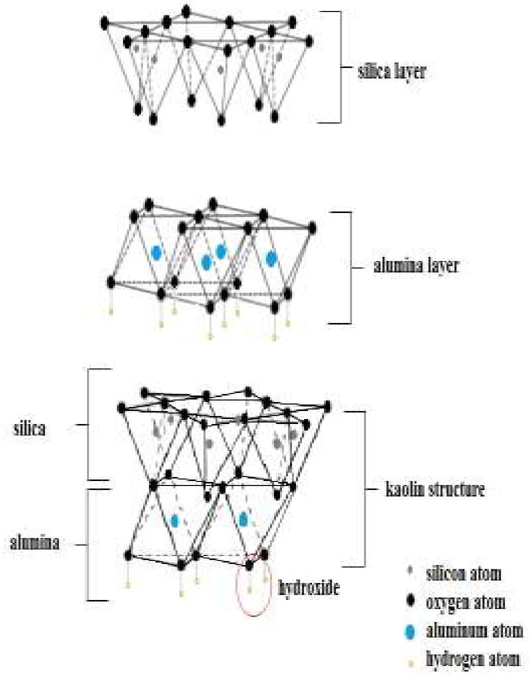

Figure 2: kaolin crystal structure

IV. Leaching with acid

Leaching 20 grams of calcined kaolin has been leached with 60 ml of Hydrochloric acid with different concentration (pH) number (0.45, 0.5, 0.55, 0.6 and 0.65) for 2 hours at different leaching temperature (30, 60 and 90) °C. The experiment materials have been stirred together for 2 hour by using magnetic stirrer in presented of glass condenser to minimize the evaporation of the acid. The silica layer is passive to the reaction with HCl so that the reaction between the acid and kaolin (alumina only) can be explained by the chemical reaction in chemical equation (1).

$6HCl\,(sol.) + Al_2O_3\,(s) \rightarrow 2AlCl_3\,(sol.) + 3H_2O$ (1)

The final product of leaching process is slurry consist of very fine silica particles which were passive in the reaction (eq. (1)) and Aluminum chloride (dissolved and undissolved) materials which can be separated by washing with distilled water four times (1litter/ 20 grams) of the clays in each time. The concentration of dissolved aluminum chloride is reduced with each washing process Figure 3.

The selection of diluted acid for this reaction is based on the nature of kaolin. The kaolin has lamellar structure consisting of one layer of each alumina and silica like a sandwich of the bilayer of (alumina and silica) with a hydrogen bond between layers figure (2). This hydrogen bond is vanishing in present of polar solvent like water; therefore the dilute acid has been used for this reaction. The water expanded the gap between the kaolin layers which allowed to the acid to react with alumina surface easily.

V. Treated with sodium hydroxide

The aluminum chloride solution has been treated with sodium hydroxide solution to precipitate aluminum hydroxide. The chemical equation (2) shows two product ($Al(OH)_3$ and NaCl) the first one is insoluble in water while the second one is soluble in water. Therefore, that physical separation has applied to separate each constituent by washing with water four times.

$AlCl_3 + 3NaOH \rightarrow Al(OH)_3 + 3NaCl$ (2)

The aluminum hydroxide dried by the electrical furnace at 200°C for 2 hours. The characterization of aluminum hydroxide has been studied by using X-ray diffraction with cu target Kα, the scanning range 10-80 (deg) with scanning rate (8 deg/min) and 40 KV applied voltage.

The XRD test gives a clear idea about the crystal structure and phases of the aluminum hydroxide. The XRD test has done in the college of education Baghdad University. The XRD charts have shown in Figure 4. The XRD test gives clear idea about the crystal structure and phases of the aluminum hydroxide.

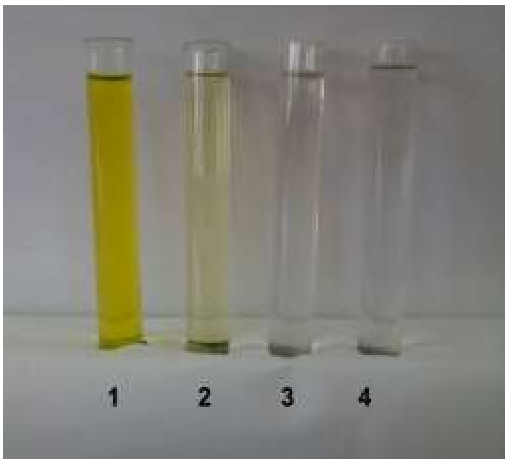

Figure 3: Physical separation between the aluminum chloride solution and silica by four times washing process with water.

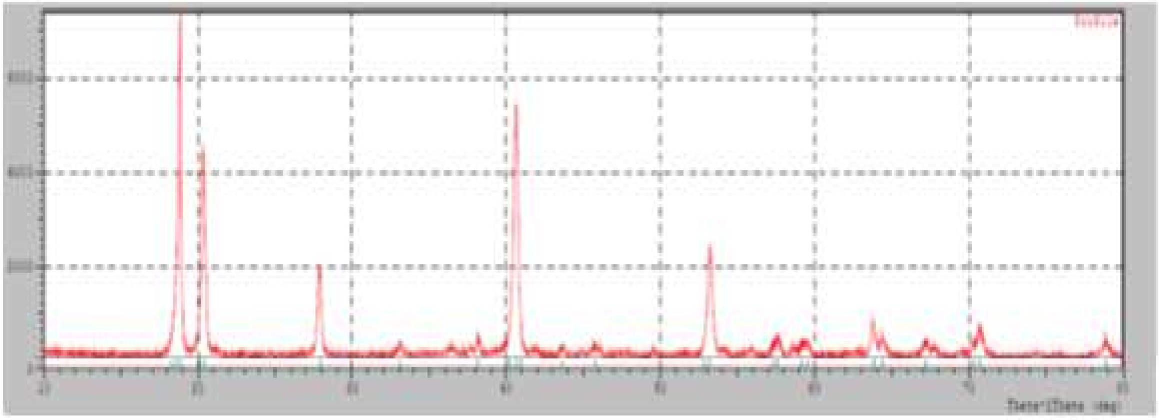

Figure 4: XRD of aluminum hydroxide

VI. Calcination of Aluminum Hydroxide

The aluminum hydroxide can be thermally converted to alumina by heating it to the temperature (1100°C) at heating rate 30 deg/min and holding for 2 hours then cooled in the furnace as shown in chemical equation (3).

$$4Al(OH)_3 \rightarrow 2Al_2O_3 + 6H_2O \qquad (3)$$

The characterization of the final calcinated alumina has studied by using X-ray diffraction with cu target Kα, the scanning range 15-85 (deg) with scanning rate (5 deg/min) and 40 KV applied voltage. The XRD test gives a clear idea about the crystal structure and phases of the aluminum oxide as shown in Figure 5. The XRD test has done in the Ministry of Science and Technology labs.

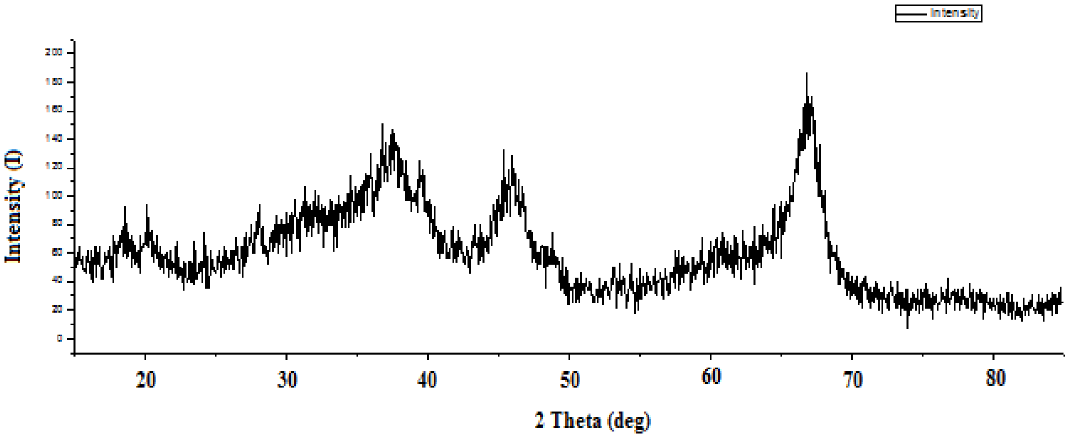

Figure 5: XRD of extracted alumina.

The chemical analysis of the final calcinated alumina has studied by using X-ray fluorescence. This test shows the chemical composition of all kind of materials with an elemental range from Sodium to Uranium. Table 3 shows the chemical composition of the final alumina, its shows the weight percent of alumina (96.98), Calcium oxide (1.3) and number of other oxides. Figure 6 shows the XRF chart for the alumina sample. This test has done in the Ministry of Sciences and Technology laboratories in Baghdad.

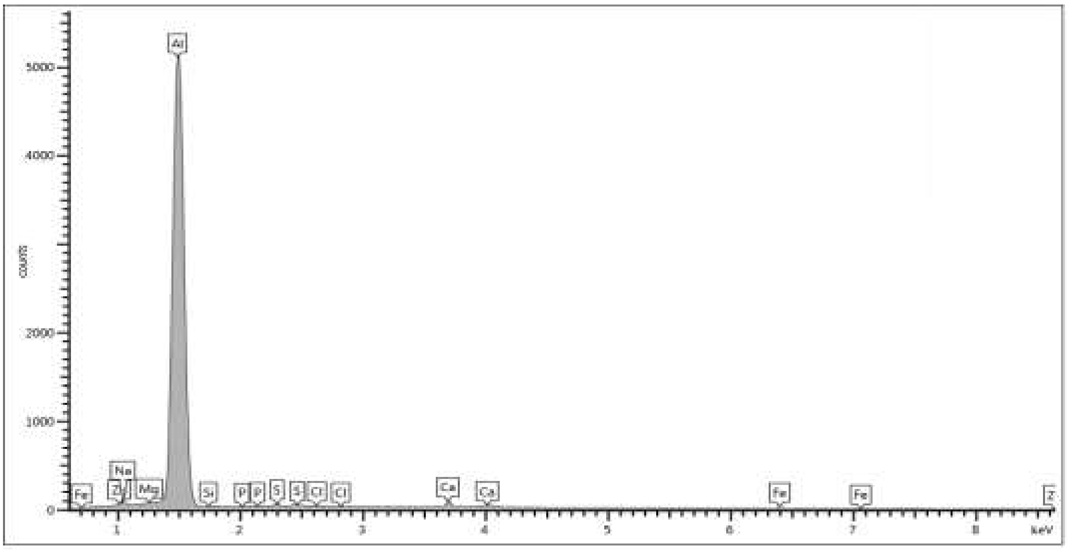

Figure 6: XRF for extracted alumina.

Table 3: chemical composition of alumina

Element	Oxide	Oxide w%
O		
Na	Na2O	0.43
Mg	MgO	0.23
Al	Al2O3	96.98
Si	SiO2	0.22
P	P2O5	0.01
S	SO3	0.07
Cl	Cl2	0.08
Ca	CaO	1.3
Fe	FeO	0.36
Zn	ZnO	0.20
Total		99.88

3 Results and Discussion

I. Chemical Analyses

The chemical analysis of the raw materials gives clear ideas regarding the amount and types of the element presented in kaolin Table 1. This chemical analysis has been determined by using X-ray diffraction for kaolin sample as shown in Figure 1. According to Table 1 most, the elements that presented in kaolin have been removed by calcination and leaching with acid that approved from the final alumina product. This method shows alumina having a cubic crystal structure with high purity exceeding 95%.

The Aluminum Hydroxide in this research has a monoclinic structure according to the X-ray diffraction with lattice parameters values (a= 5.062 Å, b= 8.671 Å, c=4.713 Å, β=90.27°), the unit cell volume equal to (206.86 Å) and finally the cryptographic density was (8.94 g/cm3). All this information is collected from (JCPDS-International Centre for Diffraction Data) Cart number (20-0011). Finally, the aluminum hydroxide is thermally converted to the alumina by heating it to the 1100 °C for two hours. The XRD and XRF tests to final alumina give clear idea about chemical composition and crystal structure of alumina. Figure 5 shows The XRD of the γ alumina has a cubic crystal structure with lattice parameter (a= 7.924 Å). All this information is collected from (JCPDS-International Centre for Diffraction Data) Cart number (29-0063). The XRF test of the alumina sample shows the elementary analysis, the aluminum was the major element compared with others, while Table 3 shows the chemical composition and weight percent of each composition.

II. Effect of leaching temperature on alumina extraction:

The leaching with acid has been studied at different temperatures which have great effect on the extraction of alumina from kaolin clay. The kaolin which is used in this reaction has been calcination at 700°C for 2 hours. The leaching temperature has been selected with only three different temperatures (30, 60 and 90) °C to give a clear idea about the effect temperature on the reaction between the kaolin and hydrochloric acid. This study shows that the extraction of alumina has been reduced with increasing the reaction temperature. This decreasing in the extraction of alumina with increasing the reaction temperature can be explained by reducing in the interaction between kaolin and hydrochloric acid due to evaporation and babbling in hydrochloric acid solution; Figure 7 shows the amount of aluminum hydroxide that extracted from kaolin at different leaching temperatures.

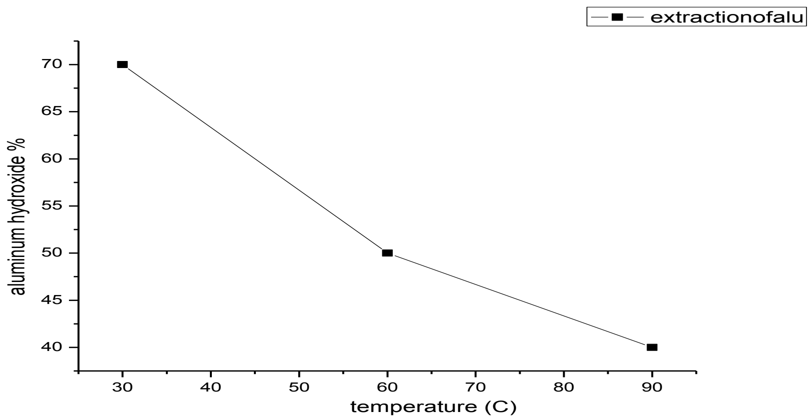

Figure 7: extraction of aluminum hydroxide at different leaching temperatures.

III. Effect of acid concentration on alumina extraction

The result that collected from the reaction between kaolin and hydrochloric acid at different pH number at fixed reaction temperature as shown in Figure 8. It indicates that the alumina extraction has increasing with decreasing the pH number of the reaction. The decreasing in the pH number refers to increase in acid concentration. The maximum extraction of alumina was recorded at the lowest pH number (0.45). The minimum extraction of alumina was recoded at the highest pH number (0.65). This result was also mentioned by [13,14]. The initial calcination temperature of kaolin for all these samples Figure 8 was 700°C.

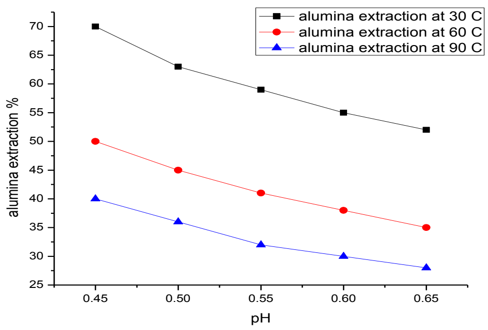

Figure 8: effect of pH number on alumina extraction at different temperatures.

IV. Effect of calcination temperature on alumina extraction

The result collected from the reaction between the kaolin which calcined at different temperature (600, 650, 700 and 750) °C and hydrochloric acid with different (pH) number as shown in Figure 9. It indicates that higher alumina extraction occurs at 700°C calcination temperature and pH number (0.45). The reaction temperature between the hydrochloric acid and kaolin for all samples figure (9) was 30°C.

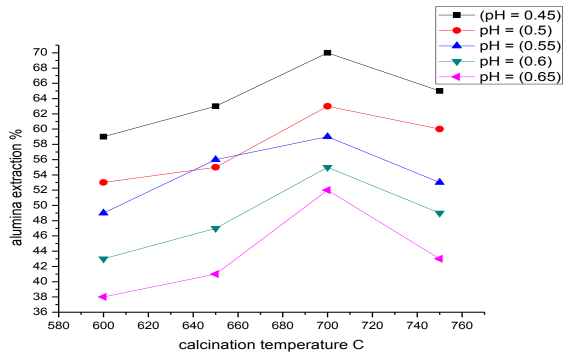

Figure 9: effect of the calcination temperature on the alumina extraction

4. Conclusions

1. Iraqi kaolin from Alduikhla astrologer having approximately 34% of alumina can be a candidate as a replacement for bauxite rack in the field production of alumina.
2. The alumina produces by this method having high purity above 95% which have many industrial applications.
3. The alumina extraction is decreased with increasing the reaction temperature due to reducing in the interaction between kaolin and hydrochloric acid, in addition to the evaporation and babbling in hydrochloric acid solution.
4. The alumina extraction is decreased with increasing the pH number of the acidic reaction due to decreasing in the acid concentration.
5. The alumina shows maximize extraction at initial calcination temperature 700°C due to the increasing in the activation of the alumina layer within the kaolin structure.
6. The reason behind the decreasing in the alumina extraction at calcination temperature

above 700°C is the kaolin structure transform from the lamellar structure to the spinal structure at high temperature according to [1].

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