

STUDY ON THE OPTIMAL LEACHING PARAMETER IN THE EXTRACTION OF SODIUM DICHROMATE FROM LOCAL CHROMITE ORE

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Abstract

The chromite ores from Thabaitgine (Mandalay Region) and West Mindon (Magway Region) were analyzed and characterized by using conventional and modern instrumental analytical techniques. It was found that the chromite ore from Thabaitgine showed higher chromium content (Cr 63.13 % of Cr₂O₃) as compared to West Mindon (Cr 41.33 % of Cr₂O₃) chromite ore. Sodium dichromate was so prepared from Thabaitgine chromite ore by leaching and pyrolysis method. The extracted compound was characterized by conventional and modern instrumental analytical techniques. The effects of raising temperature and duration of fusion time were studied to achieve maximum yield and purity. The optimal conditions, viz. the temperature at 900 °C and leaching time of 7h, resulted in the yield of 89.30 % and purity % of 95.47.

Keywords: Chromite ores, sodium dichromate, leaching and pyrolysis method, optimal yield

Introduction

Chromium occurs naturally as Iron (II) Chromite (FeCr₂O₄), Chrocoisite (PbCrO₄) and Chrome Ocher (Cr₂O₃) (Westbrook, 1979). Chromite, which is composed primarily of iron oxide and Chromium oxide, is the most important source of chromium. Chromium is the main ore of chromite which is of economic importance in Myanmar. Chromium deposits are known along in the eastern part of the Indo-Myanmar Ranges and Inner-Myanmar Tertiary Basin. Chromite ores occurs in Myitkyina Region, North Mandalay and east of the Ayeyarwady River (Hepworth, 1982). Metal chromium, its stainless steel, plating and compounds have become very important in the industrial process. Since, chromium concentrates could not easily be reduced directly to metals chromium, the intermediate compound; sodium dichromate would be a more appropriate compound to produce the metal chromium and useful chromium compounds. In order to choose the

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most appropriate prime precursor for the production of sodium chromate, chromium concentrates from different localities were firstly analyzed and characterized by using conventional and modern instrumental analytical techniques. Sodium dichromate was then extracted by leaching and pyrolysis method. It is an important precursor for the preparation of other chromium compounds (Liptort, 1980).

Materials and Methods

All the chemicals were analytical grade. Various conventional and modern instruments techniques were used throughout the experimental procedure. These include X-ray Diffractometry (XRD), Energy Dispersive X-ray Fluorescence (EDXRF), Scanning Electron Microscope (SEM) and Thermogravimetry /Differential Thermal Analysis (TG-DTA).

Sample Collection

The chromite ores from Thabaitgine Area, Mandalay Region, and West Mindon Area, Magway Region, were used in this research work. The samples were procured from No.2 Mining Enterprise, Ministry of Mines. The beneficiated samples were subjected to pretreatment process such as screening, magnetic separation, ball milling, air dried at metallurgical department and then the sample was bagged in cotton container. Sampling was carried out by cone and quartering method.

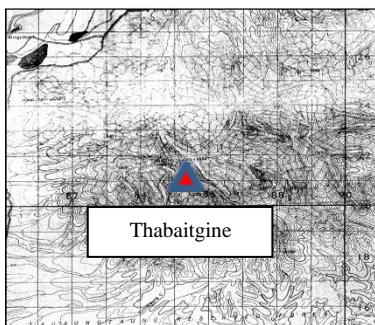


Figure 1: (a) Sampling site from Thabaitgine (Mandalay Region)

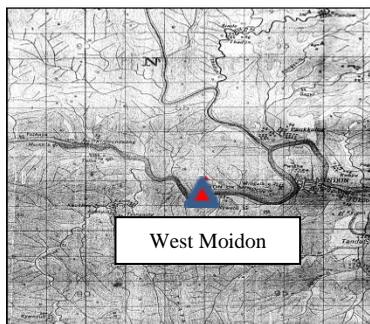


Figure 1: (b) Sampling site from West Moidon (Magway Region)

Sampling of Representative Chromite Ore Samples

Representative samples were taken systematically from a large number of materials bags. Sampling was done by piling up the well-mixed material into a cone and dividing it into quarters. Opposite quarters were rejected and the remaining half-portion again treated as before, rolling the sample back and forth on a paper. After quartering, the sample was ground in an agitate motor and pestle and then sieving with 200 mesh sieves. The representative sample was taken to determine the physicochemical analysis.

Preparation of Sodium Dichromate

Powder chromite ore (6 parts) was thoroughly mixed with (3 parts) of soda ash and (3 parts) of pyrite cinder in a porcelain crucible (Thrope, 1969). The masses were about 14.4 g chromite ore, 7.2 g soda ash and 7.2 g pyrite cinder. The contents were placed in a porcelain crucible and heated in a high temperature muffle furnace at 900 °C for 7 h. At the end of heating, the crucible was taken out and allowed to cool.

The contents in the crucible were transferred into the mortar. Then the transferred substances were crushed to powder form by means of a pestle. Again, these substances were transferred to a 500 mL beaker and hot distilled water was added into it and stirred thoroughly. The insoluble particles were allowed to settle and the yellow colour supernatant liquid was decanted into another 500 mL beaker. This procedure was repeated until the supernatant liquid became colorless. The decanted solution was filtered through filter paper to give a clear filtrate. The volume of the filtrate was reduced by evaporation. This chromate solution was found to be pH of 13.8.

The yellow filtrate was then oxidized by drop wise addition of concentrated sulphuric acid with constant stirring. KI-starch paper was prepared by soaking a strip of filter paper with 2 % starch and then with 15 % KI solution. Small pieces of the above prepared KI-starch papers and sensitive pH papers (pH 11) were placed on a glass plate to serve as external indicators in monitoring the oxidation reaction.

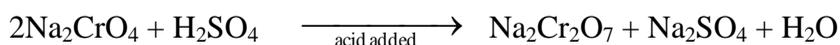
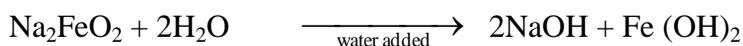
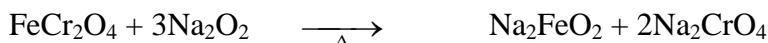
After addition of about 13 mL of concentrated sulphuric acid, the bright yellow color of the chromate solution changed to slightly orange tinge. It was tested with the KI-starch and pH papers. The color of KI-starch paper

did not change and the pH was found to be between 7 and 8. Then drop wise addition of concentrated sulphuric acid to the reaction mixture was continued with constant stirring. The solution was tested intermittently (*i.e.* after addition of 2 drops of concentrated sulphuric acid) with KI-starch and pH papers.

The oxidation was regarded to be near to completion when the color of the KI-starch paper started to change from orange (color of the reaction mixture) to brown. The pH was observed to be between 3 and 4. This reaction mixture was allowed to stand over-night in a freezer at -20 °C. The crystals deposited were removed by filtration through a Buchner funnel under reduced pressure.

The filtrate obtained above were taken in a porcelain basin and evaporated about half its volume. Then, this solution (orange in color) was further concentrated by using a thin film evaporator to get the orange crystals. Finally, almost pure sodium dichromate crystals were obtained.

The reactions of the analysis are represented by the equations:



Determination of the Optimum Reaction Time

The experimental runs were carried out at 6, 6.5, 7, 7.5 and 8 h at interval of half hour respectively. The experimental runs were made by the variation of reaction time to determine the optimum reaction time.

Determination of the Optimum Reaction Temperature

The experimental runs were carried out at 800, 850, 900 and 1000°C at intervals of 50 °C respectively. The experimental runs were made by the variation of reaction temperature to determine the optimal reaction temperature.

Property characterization

Crystal structure and phase analysis were performed by X-ray diffraction (XRD) using Rigaku, D-Max 2200, Japan. Morphology of the powder and the elemental composition were recorded by scanning electron microscope (SEM) EVO-18, ZEISS, Germany. The thermal decomposition behaviors of the sodium dichromate precursors were examined by means of thermal gravimetric and differential thermal analysis (TG-DTA) by using a Perkin Elmer. Energy dispersive X-ray fluorescence (EDXRF) confirms the elemental compositions of the prepared sample by using Shimadzu model EDX-8000 EDXRF spectrometer.

Results and Discussion

Characterization of prepared sample

XRD analysis

In this investigation, chromium concentrated samples from Thabaitgine, Mandalay Region and West Mindon, Magway Region were analyzed and characterized by using conventional and modern instrumental analytical techniques. Figures (2 a and b) confirms the intensity of distinguishing sharp resolution of the peaks in XRD patterns regarding the Thabaitgine samples being more pronounced than West Mindon samples. It was obvious that Thabaitgine sample is of a higher purity type of chromium than West Mindon sample.

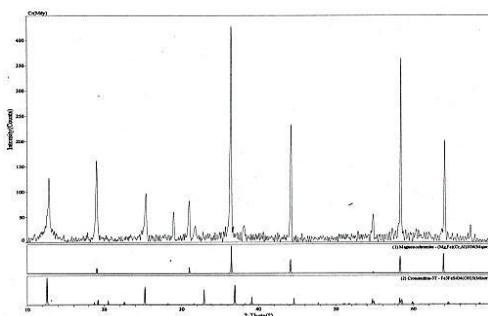


Figure 2: (a) XRD diffractogram of chromite ore from Thabaitgine (Mandalay Region)

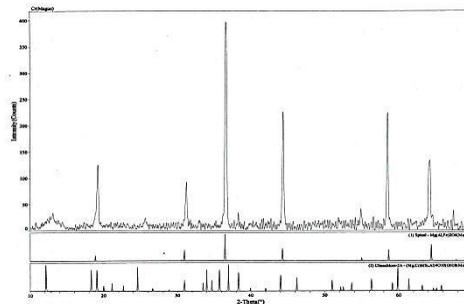


Figure 2: (b) XRD diffractogram of chromite ore from West Mindon, (Magway Region)

The relative advance of the elements in ore samples are described in Figures 3 (a and b) and then relative abundance of elements are listed in table 1. According to the experimental results, Cr and Fe as major constituents of the sample and also account to indicate that the samples contain high percentage of Cr together with trace amount of some other elements.

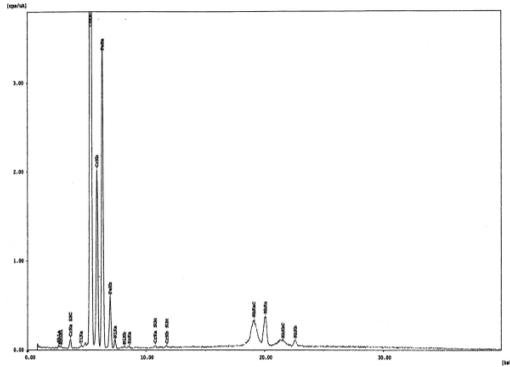


Figure 3(a) EDXRF spectrum of chromite ore from Thabaitgine (Mandalay Region)

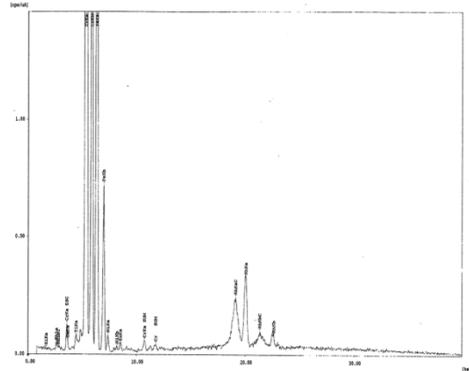


Figure 3:(b) EDXRF spectrum of chromite ore from West Mindon, (Magway Region)

Table 1: Relative Abundance of some Elements in Chromite Ore from Thabaitgine (Mandalay Region) and West Mindon (Magway Region)

No.	Elements	Relative Abundance (%)	
		Thabaitgine (Mandalay Region)	West Mindon (Magway Region)
1.	Cr	66.007	65.618
2.	Fe	33.026	31.089
3.	Ni	0.067	0.561
4.	Zn	1.023	2.173
5.	Cu	—	0.558

Analytical assay (Conventional method)

Table 2 indicates that the ore samples from Thabaitgine, Mandalay Region and West Mindon, Magway Region samples contain 63.130 and 41.330 of Cr, 16.550 and 20.400 of Si, 5.560 and 9.860 of Mg, 10.770 and 14.340 of Fe and small quantities of manganese, calcium, arsenic and lead

respectively. It can be observed that Fe, Mg, Si and Cr % of these samples nearly agree with ferberite concentrate compositions. Infact, Thabaitgine, Mandalay Region sample agree with in the range of concentrate value of that of ferberite concentrate (Smithell, 1953). On the other hand West Mindon, Magway sample show lower Cr % (i.e 41.330). The analytical data indicated that the Mandalay concentrate contains higher Cr % and less impurities than Magway concentrate. Therefore, the Mandalay concentrate was chosen as the most appropriate prime precursor for the preparation of chromium compounds in this research work.

Table 2: Analytical Assay of some Constituents Chromite Ore (Iron II Chromite) from Two Localities (Mandalay and Magway Regions)

Analyte	Weight (%)		Method of Assay
	Thabaitgine (Mandalay Region)	West Mindon (Magway Region)	
Cr ₂ O ₃	63.130	41.330	Complexometric Method
Si	16.550	20.400	Gravimetric Analysis
Mg	5.560	9.860	Gravimetric Analysis
Fe	10.770	14.340	Redox Titration
Mn	0.017	0.023	Volhard's Method
Ca	0.050	0.080	Volumetric Analysis
As	0.920	1.850	Atomic Absorption Spectroscopy
Pb	0.005	0.007	Atomic Absorption Spectroscopy
Zn	0.003	0.004	Atomic Absorption Spectroscopy
Cu	0.014	0.019	Atomic Absorption Spectroscopy
Moisture Content	0.020	0.080	Oven Drying Method

(Vogel, 1964)

Preparation of sodium dichromate

The preparation of sodium dichromate can be considered from two aspects, i.e, the variation of reaction temperature and reaction time. Table (3) and figure (4) shows the percent yield of prepared sodium dichromate with respect to fusion times at 900 °C. The data showing an optimum yield of

89.30 % was achieved corresponding to an optimum time of 7 h. The decreases in yield percentage with respect to increasing leaching time indicates that prolonged leaching time enhance the decomposition of product formed.

Table 4 and figure 5 (a) and (b) shows the percent yield and purity of sodium dichromate, prepared from chromite are at various temperature (7 hours). The yield and percent purity in the preparation of sodium dichromate with respect to the percent yield 89.30 % to the variation of the reaction temperature. The high percent yield 89.30 % and high parity 95.47 % were achieved at the optimum temperature corresponding to 900 °C.

Table 3: Percent Yield of the Prepared Sodium Dichromate with Respect to Fusion Times at 900 °C

Fusion Time (hour)	Yield (%)
6.0	54.32
6.5	68.15
7.0	89.30
7.5	78.23
8.0	66.25

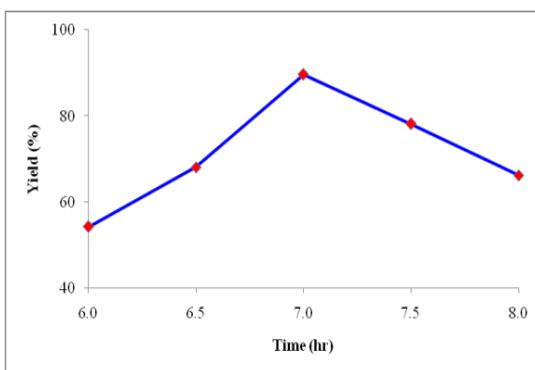


Figure 4: A plot of percent yield of prepared sodium dichromate with respect to fusion times at 900 °C

Table 4: Percent Yield and Purity of Sodium Dichromate Prepared from Chromite Ore at Various Temperatures (Fusion Time = 7 h)

Temperature (°C)	Yield (%)	Purity (%)
800	59.15	78.70
850	70.23	85.30
900	89.30	95.47
950	72.43	82.60
1000	65.85	70.90

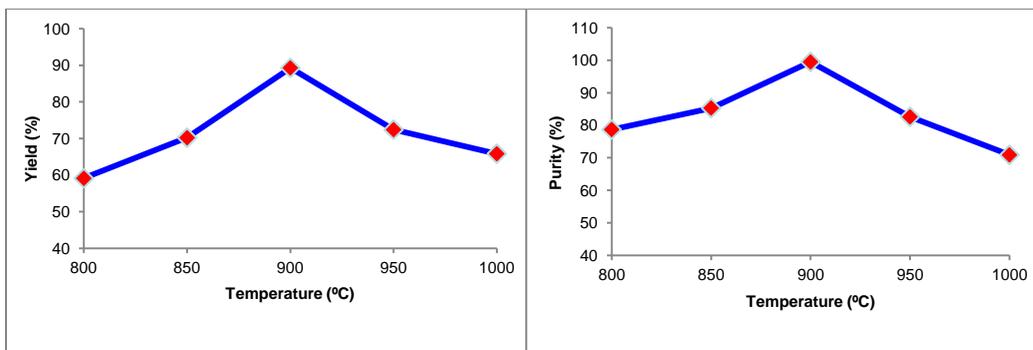


Figure 5: (a) A plot of yield percent of sodium dichromate prepared at different fusion temperatures

Figure 5: (b) A plot of purity percent of sodium dichromate with respect to different fusion temperatures

Characterization of prepared sodium dichromate

Prepared sodium dichromate was analyzed by the Analyzer Standard Method (Thorpe, 1969). It was characterized by XRD, EDXRF, SEM and TG-TDA techniques.

XRD analysis

The XRD patterns of the prepared samples sodium dichromate at different temperature are shown in figure 9. The XRD diffractogram of the prepared sodium dichromate, corresponding to optimum leaching time of 7 h at the optimum temperature of 900 °C. The XRD diffractogram patterns representing the 95.80 % of purity as shown in figure do complied with that of a specified $\text{Na}_2\text{Cr}_2\text{O}_7$ which was confirmed by the match spectrum. From XRD patterns, the prepared sodium dichromate sample also show good crystallinity.

Table 5: Relative Abundance of some Elements in Prepared Sodium Dichromate from Thabaitgine (Mandalay Region)

No.	Elements	Relative Abundance (%)
		Thabaitgine (Mandalay Region)
1.	Cr	100

SEM Analysis

The SEM micrograph of the prepared sodium dichromate is shown in figure 8. The morphology of prepared sodium dichromate is described as granules.

**Figure 8:** SEM micrograph of the prepared sodium dichromate**TG-DTA analysis**

The TG-DTA analysis of the prepared sodium dichromate is shown in figure 9 and their total weight loss are listed in Table 6. It was found the prepared samples at 108.21 °C, the temperature range 38-140°C, endothermic peak was observed and this weight loss is due to the removal of water and moisture. In the temperature range 140-200°C, An endothermic peak existing in the DTA profile of about 162.47°C may be attributed to the loss of remaining water. There is an endothermic peak of about 385.25°C, which corresponds phase change.

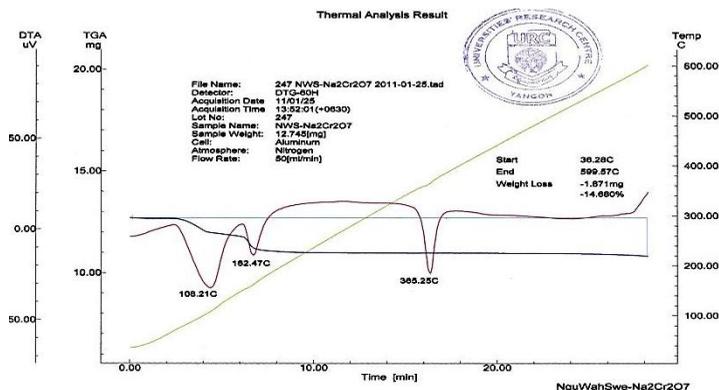


Figure 9: TG-DTA thermogram of the prepared sodium dichromate

Table 6: Thermal Analysis Data of Prepared Sodium Dichromate

TG Thermogram		DTA Thermogram Temperature (°C)	TG and DTA Remark
Break in Temperature (°C)	Weight loss (%)		
38 - 140	8.52	108.21 (Endo peak)	Endo peak due to the loss of hydrated water
140 - 200	4.26	162.47 (Endo peak)	Endo peak due to the loss of remaining water
200 - 600	2.14	385.25 (Endo peak)	Endo peak due to phase change

Conclusion

Chromite ore samples from different localities were analyzed and characterized by conventional and modern instrumental analytical techniques. The results of this investigation have shown that Thabaitgine, Mandalay Region, ore sample has higher chromium (III) oxide content and lesser amount of impurities than West Mindon, Magway Region. Thabaitgine sample contained 53.13 % of Cr, 16.55 % of Si, 5.56 % of Mg, 10.77 % of Fe and small amount of other impurities. The XRD diffractograms of the chromite ore samples are high intensity and well resolution of the peak regarding the Mandalay Region, Thabaitgine sample being more pronounced

than Magway Region, West Mindon sample. The semiquantitative EDXRF spectra of samples presented are shown in higher constituents of chromium in Thabaitgine sample being more pronounced than Magway Region. The analytical data indicated that the Mandalay Region, Thabaitgine sample contains higher Cr_2O_3 and lesser impurities than Magway Region, West Mindon sample. Therefore, the Mandalay Region, Thabaitgine sample was chosen as the most appropriate prime precursor for the preparation of chromium compounds in this research work.

Sodium dichromate has been prepared from chromite ore sample of Thabaitgine by fusion and pyrolysis method. The effects of raising the temperature and duration of fusion time were studied. The optimum conditions such as the temperature at 900 °C and leaching time at 7 h resulted in the yield of 89.30 % and purity of 95.47 % sodium dichromate. From the XRD analysis of sodium dichromate, it was found that the fine resolution diffraction peaks confirmed the highest purity of sodium dichromate. From EDXRF spectrum, the prepared sodium dichromate was semi-quantitatively evaluated. The morphology of prepared sodium dichromate is granules. Thermal analysis (TG-DTA) provides thermal stability and phase change of sodium dichromate. According to above the results, the prepared sodium dichromate is good raw material for application in industries.

Acknowledgements

The authors would like to express their profound gratitude to the Department of Higher Education, Ministry of Education, Yangon, Myanmar, for provision of opportunity to do this research and Myanmar Academy of Arts and Science for allowing to present this paper.

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