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Appendix I Cr (VI) and total chromium determination using spectrophotometer

I.1 Cr (VI) determination

I.1.1 Reagents

I.1.1.1 Colour reagent

Dissolve 0.4 g s-diphenyl carbazide (AR) in 100 ml 95% ethanol. Add 120 ml 85% phosphoric acid and dilute to 500 ml using distilled water.

I.1.1.2 Standard Cr (VI) solution

Dissolve 0.1414g $K_2Cr_2O_7$ in 1 l distilled water. Use different dilutions to prepare the calibration curve.

I.1.2 Determination procedure

I.1.2.1 Sample handling

The leaching mixture were shaken and filtered before a representative leachate sample is taken. The pH of the solution must be acidic ($pH < 2$) for the colour compound to form.

I.1.2.2 Prepare standards and calibration curves

Prepare two sets of standards (7 standards per set), ranging from 0.5 to 10mg/l (standard set 1) and from 0.02 to 0.5 mg/l (standard set 2) in 100 ml volumetric flasks. Transfer 5 ml standards solution for standard set 1 and 20 ml standards solution for standard set 2 into 25ml volumetric flasks, and pipette 3 ml of colour reagent and add distilled water to the mark. Set the solutions 20 minutes for the colour development of the mixture. Prepare a blank solution as the methods of standards just without the Cr (VI) solution. Prepare a calibration curve for the two different sets of standards at 542 nm.

I.1.2.3 Cr(VI) concentration determination

Transfer 5 ml leachate sample into 25ml volumetric flask, add 3 ml of colour reagent, and add distilled water to mark, set 20 minutes. Prepare a blank solution in exactly the same

as the leachate sample without adding 5 ml of the leachate sample. Use the calibration curve to determine the Cr (VI) concentration in the unknown sample. If the Cr (VI) concentration is higher than that in the calibration curve, the leachate sample was diluted and the Cr (VI) concentration is measured again. If the Cr (VI) concentration is lower than that in the calibration curve, prepare the sample as the lower concentrations calibration curve (standard set 2) as the following procedure: Transfer 20 ml leachate sample into 25ml volumetric flask, add 3 ml of colour reagent, and add distilled water to mark, set 20 minutes. Prepare a blank solution in exactly the same as the leachate sample without adding 20 ml of the leachate sample.

I.2 Total Cr concentration determination

I.2.1 Regents

I.2.1.1 H₂SO₄ and H₃PO₄ solution (V/V=1:1)

I.2.1.2 KMnO₄ solution (0.4%)

I.2.1.3 Urea solution (20%)

I.1.2.4 Sodium nitrite solution (2%)

I.1.2.5 Colour reagent and stock Cr (VI) standards solution (as I.1)

I.2.2. Analytical procedure

I.2.2.1 Standards preparation

Transfer 20ml standards solution for standards set of 0.02~0.5mg/l and 5ml for standards set of 0.5~15mg/l into 150ml flask and adjust pH value (approximately 7) by NH₃OH or H₂SO₄, then add 0.5ml H₂SO₄ and 0.5ml H₃PO₄ and shake. Add 4 drops KMnO₄ solution into the solution, if the purple in the solution disappear soon. Add it again to keep the purple. Vaporise the solution to 20ml.

Cool the solution down to room temperature and add 1ml urea solution. Pipette the sodium nitrite solution into the solution until the purple colour disappears. Transfer the solution to 50ml volumetric flask and dilute up to mark, and add 3 ml colour reagent, give 15 min to develop colour, use the absorption value to develop a calibration curve.

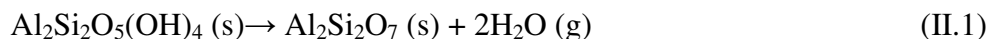
I.2.2.2 Concentration determination

The procedure is exactly same with that in I.2.2.1 only change the standards solution into the leachate samples.

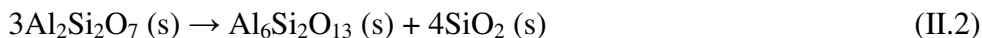
Appendix II Thermal characteristics of clays

The thermal characteristics of the clays are shown in Figure II.1 a-c, in which the weight loss (TG), differential weight loss (DTG) and differential thermal analysis (DTA) of the clays, when they are heated from room temperature up to 1300°C, are reported.

Figure II.1a shows the continuous weight loss of the clay from room temperature to approximately 900°C. The initial weight loss of the clay is mostly due to the vaporisation of the hygroscopic water in the samples (an endothermic peak at about 70°C on the DTA curve). The dehydroxylation of kaolinite at 540°C to metakaolinite, contributes to most of the weight loss (about 4%): which is associated with an endothermic peak,



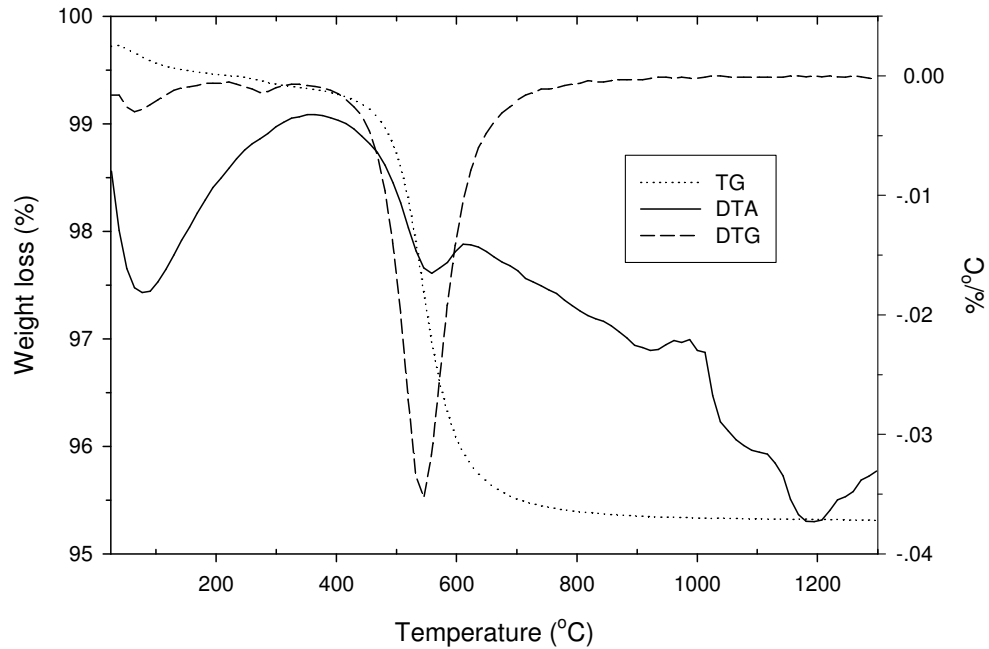
The DTA curve shows that there are several exothermic peaks at 960 °C, 980 °C, 1010 °C and 1110°C, although the weight of the sample remains stable above 900°C. According to Johnson et al. and Brindley et al., it is associated with the mullitisation of the metakaolinite [165,166], which can be expressed as:



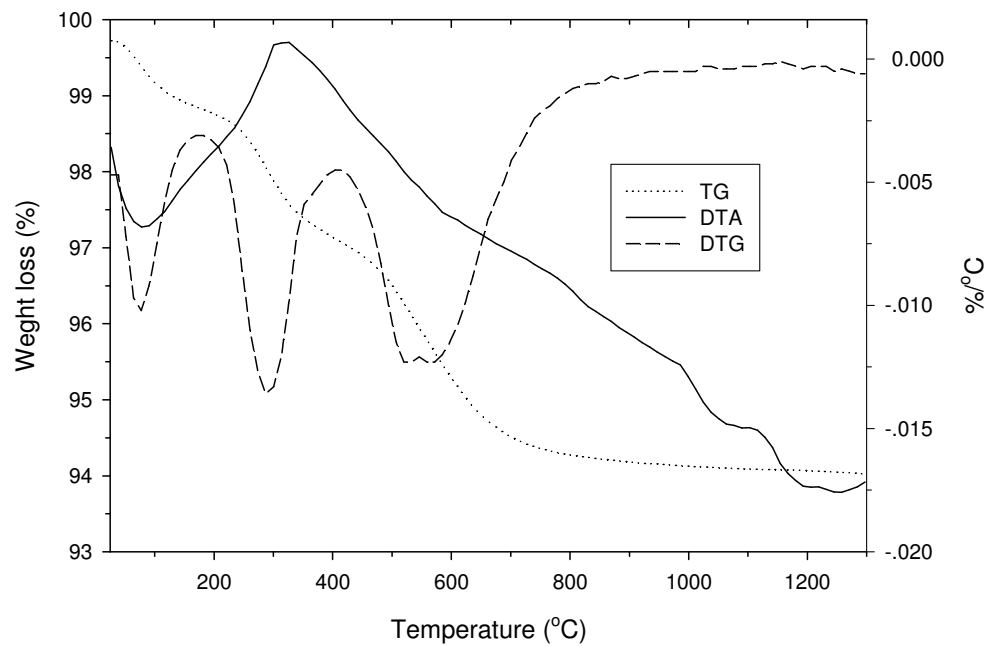
XRD analysis of the residue from the thermal experiments confirms the presence of mullite (Figure II.2). Figure II.2 also shows that cristobalite present in the residue, which is related to the polymorphic transformation of quartz during heating.

Figure II.1b shows the thermal characteristics of clay MR from room temperature to 1300 °C. The DTG curve shows that four weight loss peaks exist at approximately 75, 290, 520 and 580 °C. It is owing to the loss of absorbed water, dehydration of muscovite and dehydration of kaolinite, respectively [167-169]. The DTA curve also shows that the exothermic peaks about 980 °C and 1110°C which are associated with the mullitisation of the kaolinite in the sample.

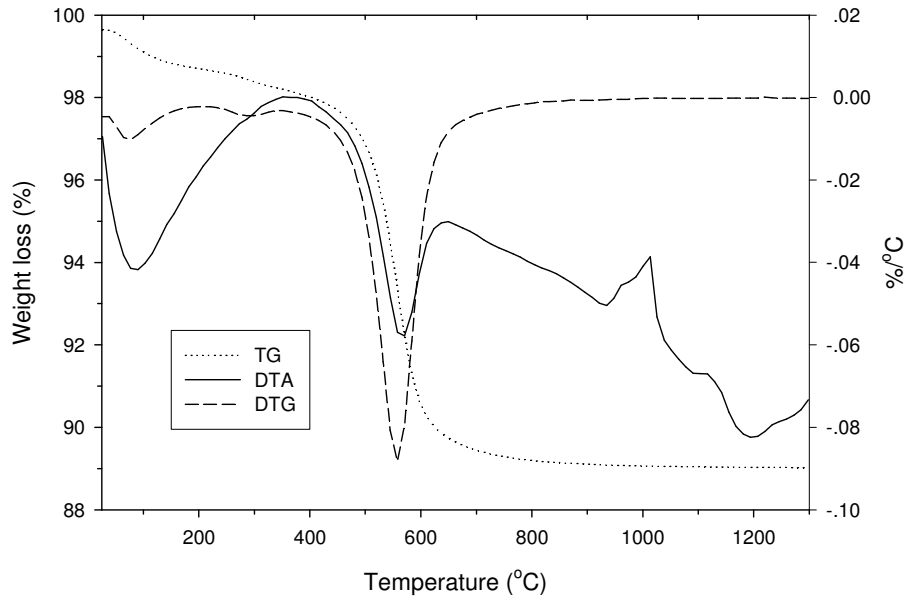
Figure II.1c shows the same tendency with clay AS. It shows the vaporisation of the absorbed water (at 90°C), dehydration of muscovite, dehydration of kaolinite (at 570°C), and the mullitisation of the sample at approximately 1000°C and 1110°C.



(a)AS



(b)MR



(c)AC

Figure II.1. The thermal characteristics of the clays (a)AS, (b)MR and (c)AC

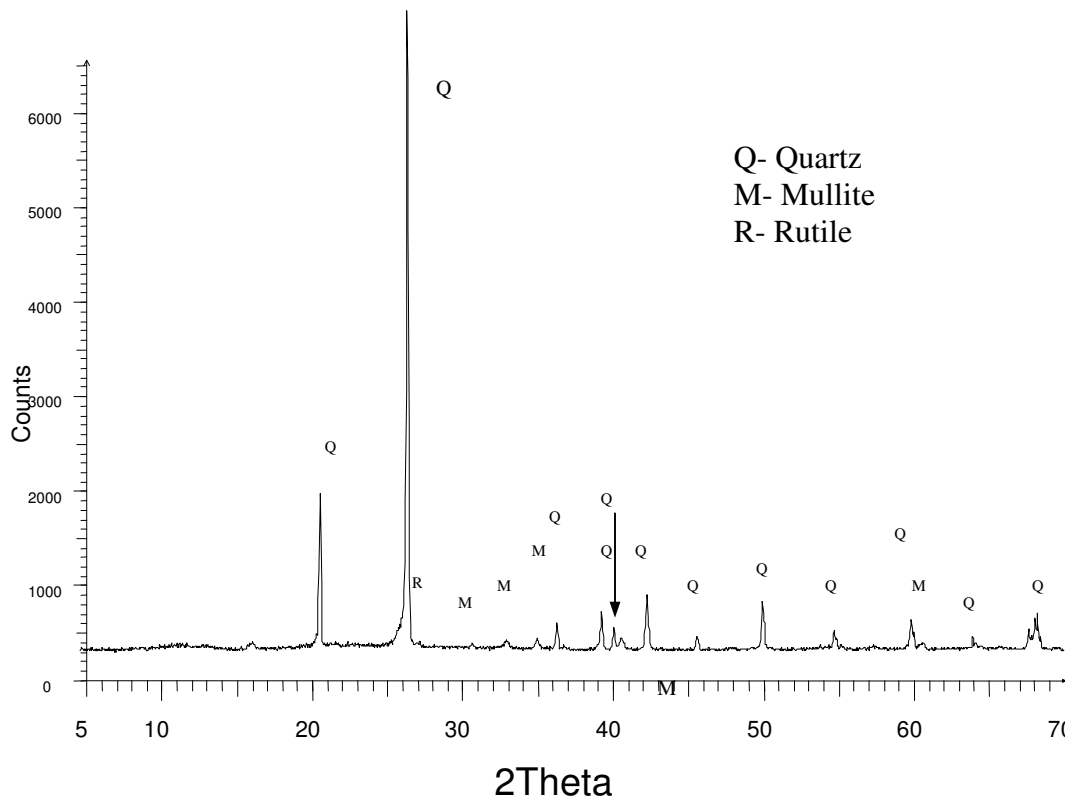


Figure II.2. XRD pattern of the residue of TG/DTA experiment (AS)

Appendix III Mass balance of the sintered brick

Table III.1 Mass balance of the synthetic sample
(AS spiked with 0.0636gCrO₃, 1200°C and 5 hours)

%	AS	AS+CrO ₃ (calculated)	Brick (analysed)
SiO₂	80.68	80.68	82.82
TiO₂	0.78	0.78	0.81
Al₂O₃	12.20	12.20	12.68
Fe₂O₃	0.99	0.99	1.08
MnO	0.02	0.02	0.00
MgO	0.06	0.06	0.02
CaO	0.00	0.00	0.00
Na₂O	0.06	0.06	0.03
K₂O	0.87	0.87	0.9
P₂O₅	Nd	Nd	0.02
Cr₂O₃	0.02	0.117	0.095
NiO	Nd	Nd	0.00
V₂O₅	Nd	Nd	0.01
PbO	Nd	Nd	0.002
LOI	3.84	3.84	0.03
Total	99.45	99.55	98.50

Table III.2 Mass balance of the sintered 50%AS-50%SPD (1100°C and 5 hours)

%	SPD	AS	50%AS-50%SPD (calculated)	Brick (analysed)
SiO₂	4.81	80.68	43.04	43.53
TiO₂	0.08	0.78	0.43	0.49
Al₂O₃	0.40	12.20	6.34	7.17
Fe₂O₃	43.4	0.99	24.52	23.96
MnO	5.08	0.02	2.59	2.73
MgO	5.44	0.06	2.79	2.28
CaO	12.9	0.00	6.55	6.12
Na₂O	0.60	0.06	Nd	Nd
K₂O	0.97	0.87	0.93	0.73
P₂O₅	0.04	Nd	Nd	Nd
Cr₂O₃	14.6	0.02	7.42	7.86
NiO	2.79	Nd	1.42	1.80
V₂O₅	0.09	Nd	Nd	0.06
ZnO	4.49	Nd	2.28	2.14
MoO₃	1.35	Nd	Nd	Nd
PbO	0.39	Nd	Nd	0.06
LOI	-0.21	3.84	1.82	0.07
Total	98.55	99.45	100.13	99.01

Table III.3 Mass balances of the sintered bricks that contained 50%AS and 50% ferrochrome dust or filter cake (1000°C and 5 hours)

%	50AS+50FCD1 (calculated)	Brick (analysed)	50AS+50FCD2 (calculated)	Brick (analysed)	50AS+50FC** (calculated)	Brick (analysed)
SiO ₂	57.59	61.14	60.69	63.08	41.41	47.03
TiO ₂	0.44	0.47	0.43	0.46	0.42	0.48
Al ₂ O ₃	8.55	8.84	8.09	8.52	6.44	7.35
Fe ₂ O ₃	1.86	1.91	1.60	1.67	10.28	11.28
MnO	0.22	0.21	0.27	0.28	0.49	0.64
MgO	5.76	6.03	9.56	9.97	0.62	0.46
CaO	0.25	0.25	0.19	0.14	19.38	22.92
Na ₂ O	4.96	4.18	3.29	2.26	0.20	0.26
K ₂ O	1.71	2.57	2.39	2.93	0.45	0.50
P ₂ O ₅	0.01	0.02	0.01	0.02	0.02	0.04
Cr ₂ O ₃	1.59	1.67	1.72	1.80	1.55	1.70
NiO	0.01	0.01	0.01	0.01	0.73	0.80
V ₂ O ₅	0.01	0.02	0.01	0.02	0.01	0.02
ZnO	7.30	7.99	4.78	4.65	0.20	0.12
Cl*	1.60	0.84	0.47	0.03	0.03	0.01
SO ₃ *	1.63	3.98	1.18	0.88	3.55	1.6
F*	0.39	0.29	0.02	0.01	11.01	10.54
LOI	5.98	1.60	5.16	0.95	8.87	3.54
Total	99.86	102.2	99.87	97.68	105.66	109.29

: Elements that are indicated with an “” should be considered semi-quantitative;

** : It is assumed that calcium in the filter cake is present as CaF₂, the total content of the filter cake is therefore not calculated using 113.49% but 102.18% (Table 3.2).

Appendix IV Calculations on the acceptable Cr (VI) concentration in the leachate

Due to the fact that the stainless steel plant can generate approximately 2,000 t stainless steel dust monthly, the total amount of bricks that can be produced by mixing stainless steel plant dust and clay would amount to $\frac{2 \times 10^8}{R_{SPD}}$ (kg/month) by assuming that all the stainless steel dust are treated, where R_{SPD} is the ratio of stainless steel plant dust in the brick (%).

Since the mass of one sintered brick is approximately 125 grams and it has a dimension of approximately 28mm×20mm×100mm, the surface area of one sintered brick can be calculated as:

$$\begin{aligned} \text{Surface area (m}^2\text{)} &= 2 \times (28 \times 20 + 20 \times 100 + 28 \times 100) \times 10^{-6} \\ &= 0.01072 \text{ m}^2 \end{aligned}$$

The quantity of the stabilised stainless steel plant dust bricks which can be made from these wastes is $\frac{2 \times 10^8}{R_{SPD} * 0.125}$.

Thus the total surface area of these bricks is:

$$\text{Total surface area (ha/month)} = 0.01072 \times 10^{-4} \times \frac{2 \times 10^8}{R_{SPD} * 0.125} \text{ (ha/month)}$$

Furthermore, the amount of Cr (VI) in one sintered brick can be calculated as:

The amount of Cr (VI) in one sintered brick (mg)

$$\begin{aligned} &= C_{Cr(VI)} \times \frac{\text{volume of the leachate (l)}}{\text{mass of the sintered brick in one leaching test (kg)}} \times \text{mass of one brick (kg)} \\ &= C_{Cr(VI)} \text{ (mg/l)} \times \frac{0.81}{0.04 \text{ kg}} \times 0.125 \text{ kg} \end{aligned}$$

Where $C_{Cr(VI)}$ is the Cr (VI) concentration in the leachate (mg/l).

Assuming that the total area of the dumping site is the same as the total surface area of the bricks, the total amount of Cr (VI) on this site per month would be:

Total Cr (VI) load on the dumping site (mg/month)

$$= C_{\text{Cr(VI)}} \times \frac{0.8}{0.04} \times 0.125 \times \frac{2 \times 10^8}{R_{\text{SPD}} * 0.125}$$

Then the EEC value can be calculated using the following function [15]:

$$\text{EEC (ppb)} = \text{dose (g/ha/month)} \times 0.66^1$$

$$= \left[C_{\text{Cr(VI)}} \times \frac{0.8}{0.04} \times 0.125 \times 10^{-3} \times \frac{2 \times 10^8}{R_{\text{SPD}} * 0.125} \right] / \left[0.01072 \times 10^{-4} \times \frac{2 \times 10^8}{R_{\text{SPD}} * 0.125} \right] \times 0.66$$

$$= 1539.2 C_{\text{Cr(VI)}} \text{ (ppb)}$$

The stainless steel plant dust containing bricks can only be used when the EEC is lower than 0.02 ppm (20ppb). Therefore,

$$\text{EEC (ppb)} = 1539.2 C_{\text{Cr(VI)}} < 20 \text{ ppb}$$

That is, the Cr (VI) concentration in the leachate ($C_{\text{Cr(VI)}}$) should be less than 0.013 mg/l.

¹⁾ 0.66 is calculated from the ratio of the toxic substance in a weight of underground body of water [15].

Appendix V The production process of synthetic calcium chromate

The process whereby CaCrO_4 was prepared is similar to that described by Clark et al. [198]. 30 g of lime (AR grade) was slowly added into 59.6 ml hydrochloric acid (AR grade, 32% and 1.15g/ml) with vigorous stirring using a glass rod. The solution was heated to boil. 100 ml Na_2CrO_4 solution which was made by dissolving 70.2 g $\text{Na}_2\text{CrO}_4 \cdot 2\text{H}_2\text{O}$ in distilled water (approximately 3 mol/l) was then added in the solution slowly with vigorous stirring. The mixture was stirred for about 1 hour. The obtained precipitate was washed with boiling water 6 times and filtered using filter paper. It was then dried at 110°C for 18 hours, after which it was heated to 400°C for 2 hours. The powder product was examined using XRD. The XRD pattern confirmed that the product is calcium chromate (Figure V.1).

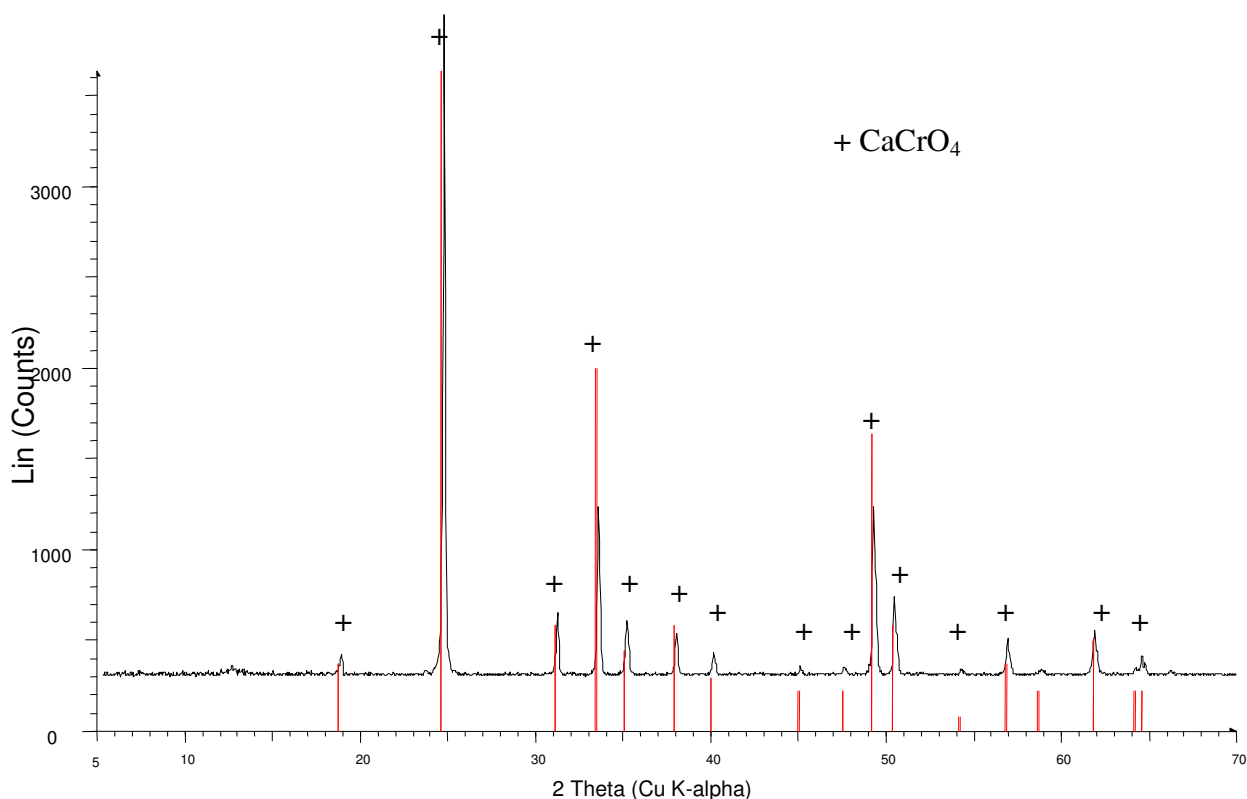


Figure V.1 The XRD pattern of calcium chromate

**Appendix VI Details of experiments on leaching behaviour of
chromium from the stabilised wastes**

Table VI.1 Average experimental values for the concentrations of Cr (VI) and Cr (T)
in the leachate

Cumulative leach time (h)	Cr (VI) (mg/l)					Cr (T) (mg/l)				
	M1	M2	M3	M4	M5	M1	M2	M3	M4	M5
1	0.275	0.025	0.004	0.052	0.144	0.275	0.026	0.007	0.055	0.144
9	0.581	0.115	0.014	0.108	0.505	0.583	0.120	0.006	0.112	0.496
25	0.361	0.171	0.014	0.058	0.441	0.371	0.179	0.007	0.057	0.492
49	0.157	0.090	0.007	0.013	0.571	0.155	0.085	0.006	0.008	0.579
81	0.049	0.026	0	0.001	0.522	0.047	0.026	0	0	0.538
121	0.027	0.006	0	0	0.419	0.015	0.037	0	0	0.521
169	0.030	0.002	0.006	0.003	0.474	0.046	0.013	0.014	0.013	0.472
225	0.009	0	0	0	0.403	0.043	0.027	0.028	0.047	0.418
289	0.009	0	0	0	0.427	0.019	0	0	0.012	0.329
361	0.035	0.025	0.008	0.008	0.398	0.026	0	0.008	0.002	0.407
441	0.015	0	0.001	0	0.4	0.017	0	0.002	0	0.419
529	0.013	0.001	0.002	0	0.389	0.021	0.001	0.007	0	0.376
625	nd	nd	nd	nd	0.365	nd	nd	nd	nd	0.367
729	0.035	0.016	0	0.002	0.317	0.038	0.004	0.002	0.008	0.309
961	0.031	0.001	0	0	0.452	0.030	0.022	0	0	0.442
1225	nd	nd	nd	nd	0.305	nd	nd	nd	nd	0.300
1521	0.045	0.002	0.002	0	0.17	0.042	0	0	0	0.199
1849	0.037	0.006	0.003	0.005	0.068	0.018	0	0.001	0	0.062
2209	0.025	0	0.002	0	0.037	0.032	0	0	0	0.047
2601	0.042	0.003	0.002	0	nd	0.048	0.006	0.001	0.001	nd
3025	0.037	0	0.004	0.003	nd	0.041	0.023	0	0	nd
3481	0.053	0.007	0.009	0.007	nd	0.040	0.012	0.006	0.005	nd

Note:nd-not determined.

Experimental conditions:

Mass of the sample: ~ 40g

Shape of the sample: Cylinder ($\Phi 25\text{mm} \times 40 \pm 0.36\text{mm}$)

Furnace: Muffle furnace

Sinter temperature: 1100°C for 50%SPD+50%AS (M1) and 50%SPD+50%MR (M2), 1000°C for 20%FCD1+80%AS (M3), 20%FCD2+80%AS (M4) and 20%FC+80%AS (M5)

Sinter time: 5 hours

Heating rate: ~4°C/min

Cooling rate: ~1.6°C/min

Leaching temperature: 25°C