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Developmental Process Study for Laboratory Scale Preparation of Barium Chromate

¹Sheikh Asrar Ahmad, ¹Maria Mustafa, ¹Nafeesa Batool, ¹Asad Gulzar, ¹Sajid Mahmood Rao, ¹Qamar Ali, ²Muhammad Nadeem Zafar, ³Adnan Akram, ⁴Rana Khalid Mahmood, ⁴Zia Ullah Khokhar and ⁵Muhammad Abdul Qadir

¹Division of Science and Technology, University of Education, Lahore, Pakistan

²Department of Chemistry, University of the Gujrat, Pakistan

³PCSIR Laboratories complex, Feroz Pur Road Lahore, Pakistan

⁴Government Post graduate Islamia College Gujranwala, Pakistan

⁵Institute of Chemistry, University of the Punjab Lahore, Pakistan

Abstract: Present work deals with the developmental process study for laboratory scale preparation of barium chromate. It is a cheaper and a viable method to prepare barium chromate which has very important industrial applications. Barium chromate was prepared by mixing different concentrations of sodium chromate and barium chloride. Percentage yields of barium chromate were determined 86%, 93%, 91%, 85% and 90% respectively. Different parameters such as temperature, *pH*, stirring speed, solubility were applied to get optimum yield. The optimum percentage yield was noted 93% for 0.3 M concentrations of sodium chromate and barium chloride at *pH* 7, 25°C without stirring. Barium was estimated gravimetrically in sample solution as barium sulfate and its optimum percentage yield was 46%. Percentage of chromium estimated by atomic adsorption spectrometry in sample solution was found to be 19.5%. The purity of barium chromate was checked by iodine titration method and maximum purity of sample solution was found to be 98%.

Key words: Barium chloride • Barium chromate • Sodium chromate • Laboratory scale

INTRODUCTION

 ${\rm BaCrO_4}$ is considered to be genotoxic and cytotoxic [1] the other positive side of the picture in extensive applications in some industries as a pigment in paints, ceramics, coloring glasses, fuses and porcelains. It is used as a corrosion inhibitor to prevent electrochemical corrosion at the joints of dissimilar metals, safety matches, metal primers and in ignition control devices. It is also used as an initiator for explosives and vapor-phase oxidation catalyst [2-10].

Various researchers have studied the laboratory scale preparation of barium chromate. For example Firsching [11] used complex formation and replacement methodology to precipitated BaCrO₄ from a homogeneous solution. De Silva *et al* [12] studied the rate of the crystal growth of barium chromate using the technique of precipitation from homogeneous solution (PFHS), by means of urea hydrolysis. The experimental conditions

which were varied included rate of change of solubility, operating temperature and stirrer speed. Guangjun *et al.* [13] explored that BaCrO₄ nano particles with uniform size and morphology were synthesized successfully via a simple wet chemical process by using a surfactant. Zhonghao [14] and coworkers prepared well-defined super structures of rectangular-shaped BaCrO₄ and network of BaCrO₄ nano particles constructed by self-assembly in Acetyl tri methyl ammonium bromide (CTAB) reverse micro emulsions. Toshiyuki N. *et al.* [15] studied the shape and size control of barium chromate nano particles using reverse microlle.

In the present study, a process was developed for the laboratory scale preparation of barium chromate. BaCrO₄ crystallized as light yellow transparent rhombic crystals which were isomorphous with barium sulfate as reported in UllMann's Encyclopedia [16]. Percentage purity of Barium Chromate was determined by using AAS. The effect of other parameters such as concentration of

Corresponding Author: Sheikh Asrar Ahmad, Division of Science and Technology, University of Education, Lahore, Pakistan. Cell: +92-333-8255310.

reactants, temperature, pH and stirring speed was also determined to know optimum conditions. Optimum percentage yield of BaCrO₄ was obtained using 0.3 M sodium chromate and barium chloride at pH= 7, 25°C and without stirring. Present work is very less expensive and a viable method as compared to reported methods of preparation of barium chromate.

MATERIALS AND METHODS

Glass Ware: All used glassware made of Pyrex. Graduated cylinder (100 mL, 500 mL, 1000 mL), Beakers (200 mL, 500 mL, 1000 mL), Pipette (1 mL, 5 mL, 10 mL, 20 mL), Separating funnel, Conical flasks (250 mL), Volumetric flasks, Gooch crucible, Glass rod, Funnel.

Chemicals Required: All chemicals were used of analytical grade. Sodium hydroxide, Hydrochloric acid, Acetic acid, Barium chloride, Sodium chromate, Sulfuric acid, Nitric acid, Sodium thiosulfate, Potassium iodide, Starch.

Instruments Used: Electrical Stirrer, Analytical Electric Balance, Electric Oven, Atomic Absorption Spectrophotometer.

Methodology: Different amounts of barium chloride and sodium chromate were mixed in de-ionized water. A yellow precipitate of barium chromate was formed along with soluble sodium chloride salt. Percentage purity of Barium Chromate was determined by using AAS. The effect of other parameters such as concentration of reactants, temperature, pH and stirring speed was also determined to know optimum conditions. Estimation of Ba and Cr were also determined using standard method.

RESULTS AND DISCUSSION

Selected Method of Preparation: When an aqueous solution of barium salt or baryta water is treated with alkali mono or dichromate, then a pale yellow ppt. of $BaCrO_4$ is deposited. Barium chromate can be prepared by different methods.

Method No.1: Aqueous solution of potassium chromate reacts with aqueous solution of barium chloride to produce BaCrO₄(pale yellow precipitates), e.g.

 $K_2CrO_4 + BaCl_2 \rightarrow 2KCl + BaCrO_4$

Method No.2: When an aqueous solution of potassium chromate reacts with an aqueous solution of barium nitrate, then a pale yellow precipitates of BaCrO₄ is formed e.g.

 $K_2CrO_4 + Ba (NO_3)_2 \rightarrow 2KNO_3 + BaCrO_4$

Method No.3: An Aqueous solution of sodium chromate reacts with an aqueous solution of barium chloride to produce a pale yellow BaCrO₄ precipitates

e.g. Na₂CrO₄ + BaCl₂ - 2NaCl + BaCrO₄

Weighed amount of barium chloride was added to 100 mL of de-ionized water to prepare aqueous solution of barium chloride and then weighed amount of sodium chromate was added to 100 mL of de-ionized water to make aqueous solution of sodium chromate. Both of the aqueous solutions were mixed and yellow precipitates of barium chromate were formed along with soluble sodium chloride salt. Precipitates of barium chromate were filtered and dried in the oven.

Method No. 3 was selected for further experiments due to easily availability of required reactants.

Purity Test for Barium Chromate: About 1.0 g barium chromate was taken and dried for 18 hours over H₂SO₄. Loss in the weight was not more than 1.0 %. About 0.30 g barium chromate was taken from dried sample and transfer it to a glass stoppered flask, added 50 ml H₂O, 3.0 g of KI and 7.0 mL HCl. Flask was allowed to stand for 15 minutes in the dark with frequent shaking then diluted it to 75 ml of H₂O and titrated the liberated iodine with 0.1 N sodium thiosulfate using starch towards the end point. Three concordant readings were taken and purity was found 97.12%, 98.25% and 98.53 % respectively.

Effect of Concentration on the Percentage Purity of Barium Chromate

Effect of 0.1 M Concentration: 0.1 M solution of Barium Chloride (0.208 g / 100 mL) and Sodium Chromate (0.162 g / 100 mL) were prepared respectively. Mixed both of the aqueous solutions and yellow precipitates of barium chromate was formed along with soluble sodium chloride salt. Filter the precipitate of barium chromate and dried in oven and calculation was made for percentage yield. Following calculations were performed, Weight of filter paper = W1 = 1.0g, Weight of filter paper + wt. of crystals = W2 = 1.22g,

Weight of crystals = W3 = 1.22g - 1g = 0.22g and Theoretical yield = 0.253g, Practical yield = 0.22g, %age yield = 86.0%

Effect of 0.3 M Concentration: 0.3 M solution of Barium Chloride (0.62g / 100 mL) and Sodium Chromate (0.49 g / 100 mL) were prepared respectively. Both of the aqueous solutions were mixed and yellow precipitates of barium chromate were formed along with soluble sodium chloride salt. Precipitates of barium chromate were filtered and dried in an oven and calculations were made for percentage yield. Following calculations were performed, Weight of filter paper = W1 = 1.0g, Weight of filter paper + wt. of crystals = W2 = 1.71g, Weight of crystals = W3 = 1.71g - 1g = 0.71g and Theoretical yield=0.76g, Practical yield=0.71g, %age yield=93.0%

Effect of 0.5 M Concentration: 0.5 M solution of Barium Chloride (1.04g/100 mL) and Sodium Chromate (0.81 g/100 mL) were prepared respectively. Both of the aqueous solutions were mixed and yellow precipitates of barium chromate were formed along with soluble sodium chloride salt. Precipitates of barium chromate were filtered then dried in oven and calculation was made for percentage yield. Following calculations were performed, Weight of filter paper = W1 = 1.0 g, Weight of filter paper + wt. of crystals = W2 = 2.15g, Weight of crystals = W3 = 2.15g - 1g = 1.15g and Theoretical yield = 1.26g, Practical yield = 1.15g, %age yield = 91.0%

Effect of 1.0 M Concentration: 1.0 M solution of Barium Chloride (2.08g / 100 mL) and Sodium Chromate (1.62 g / 100 mL) were prepared respectively. Both of the aqueous solutions were mixed and yellow precipitates of barium chromate were formed along with soluble sodium chloride salt. Precipitate of barium chromate were filtered

then dried in oven and calculation was made for percentage yield. Following calculations were performed, Weight of filter paper = W1 = 1.0g, Weight of filter paper + W1 = 1.0g, Weight of crystals = W2 = 3.17g, Weight of crystals = W3 = 3.17g - 1g = 2.17g and Theoretical yield = 2.53g, Practical yield = 2.17g, %age yield = 85.0%

Effect of 1.5 M Concentration: 1.5 M solution of Barium Chloride (3.12g/100 mL) and Sodium Chromate (2.43 g/100 mL) were prepared respectively. Both of the aqueous solutions were mixed and yellow precipitates of barium chromate were formed along with soluble sodium chloride salt. Precipitate of barium chromate were filtered then dried in and calculation was made for percentage yield. Following calculations were performed, Weight of filter paper = W1 = 1.0g, Weight of filter paper + wt. of crystals = W2 = 4.44g, Weight of crystals = W3 = 4.44g - 1g = 3.44g and Theoretical yield = 3.80 g, Practical yield = 3.44g, %age yield = 90.0%.

Different molar concentration solutions were made to check the optimum percentage yield of barium chromate, which was obtained from 0.3 molar concentrations as shown in Figure 1.

Percentage yield of barium chromate was determined as shown in Table 1.

Effect of Temperature on Percentage Yield of Barium

Chromate: To investigate the effect of temperature on barium chromate prepared 0.3 M solution of barium chromate and diluted to 200 mL distilled water and divided into five equal parts, the temperature was maintained as 40°C, 50°C, 60°C, 70°C and 80°C respectively, the stirring speed also was kept constant and the percentage yield was calculated. The effect of temperature on percentage yield at constant stirring had been shown in Table 2 and Figure 2.

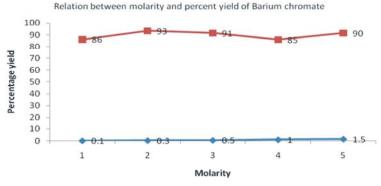


Fig 1: Effect of Molarity on Percentage yield of barium chromate

Table 1: Effect of the Molarity on Percentage yield of barium chromate

Batch No.	Molarity	BaCl ₂ (g)	Na ₂ CrO ₄ (g)	BaCrO ₄ (g)	% age yield
1	0.1	0.208	0.162	0.22	86
2	0.3	0.62	0.49	0.71	93
3	0.5	1.04	0.81	1.15	91
4	1.0	2.08	1.62	2.17	85
5	1.5	3.12	2.43	3.44	90

Table 2: Effect of temperature on percentage yield at constant stirring

Temperature°C	Actual Yield (g)	Theoretical Yield (g)	Percent yield (%)
25	0.14	0.15	93
40	0.13	0.15	86
50	0.12	0.15	80
60	0.10	0.15	66
70	0.11	0.15	73
80	0.09	0.15	60

Table 3: Effect of stirring speed on % age yield at 25°C

Stirring Speed (RPM)	Actual Yield (g)	Theoretical Yield (g)	Percent Yield (%)
20	0.13	0.15	86%
30	0.142	0.15	94%
40	0.12	0.15	80%
50	0.1	0.15	66%
60	0.11	0.15	73%

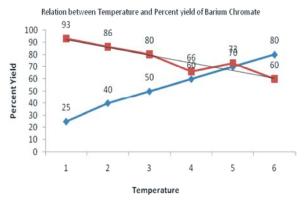


Fig 2: Effect of temperature on percent yield of barium chromate

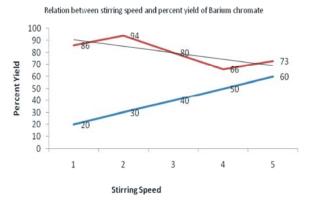


Fig 3: Effect of stirring speed on percent yield of barium chromate

Effect of Stirring Speed on Percentage Yield of Barium

Chromate: To investigate the effect of stirring speed on barium chromate prepared 0.3M solution of barium chromate and diluted to 200 mL distilled water and divided into five equal parts, the stirring speed was maintained as, 20 rpm, 30 rpm, 40 rpm, 50 rpm, 60 rpm respectively, the temperature also was kept constant and the percentage yield was calculated. The effect of stirring speed on percentage yield at constant stirring had been shown in the Table 3 and Figure 3.

Effect of pH On Percentage Yield of Barium Chromate:

To investigate the effect of pH on barium chromate prepared 0.3M solution of barium chromate and diluted to 200 mL distilled water and divided into five equal parts, the stirring speed and the temperature were also kept constant and the percentage yield was calculated. The effect of pH on percentage yield at both constant temperature (25°C) and stirring had been shown in Table 4 and Figure 4. The same range of pH has been reported by Upadhyaya K.N [18] during Step wise precipitation of barium sulfate and chromate gravimetrically.

Solubility Barium Chromate: Barium chromate is insoluble in distilled water, cold distilled water, hot distilled water and acetic acid, Soluble in hydrochloric acid in slightly warming conditions, Soluble in sulfuric

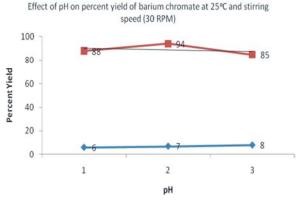


Fig 4: Effect of pH on percent yield of barium chromate

Table 4: The effect of pH on the % age yield of Barium Chromate at 25°C and 30 rpm.

рН	Practical Yield (g)	Theoretical Yield (g)	% yield
6	0.132	0.15	88%
7	0.142	0.15	94%
8	0.128	0.15	85%

Table 5: Solubility of BaCrO₄ in water and acids

Solvent	Observations
Distilled water	In soluble
Cold distilled water	In soluble
Hot distilled water	In soluble
Acetic acid	In soluble
Hydrochloric acid	Soluble
Sulfuric acid	Soluble
Nitric acid	Soluble

Table 6: Solubility behavior of BaCrO4 in different percent solutions of HCl

HCl solution %	BaCrO _{4 dissolved (g)}	Water	pН
1%	0.14	Yes	3
3%	1.19	Yes	3
5%	2.64	Yes	5

acid in slightly warming conditions, nitric acid in slightly warming conditions. The effect of solubility on percentage yield at constant stirring had been shown in Table 5.

Solubility behavior of BaCrO₄ in different percentage solutions of HCl: To check out the solubility behavior of barium chromate percent solution of hydrochloric acid of different percentages were prepared (1%, 3%, 5%, 7%, 10%) respectively and dissolved the barium chromate in each solutions until the no more amount of barium chromate dissolved in hot solution. After the completion of every step pH of solution was noted. The solubility behavior of barium chromate in different percentage solutions of HCl had been shown in Table 6.

Chromium Estimation: 0.5g of BaCrO₄ was taken in 250 mL flask, added 2.0 mL of HCl and few drops of conc. HNO₃ and diluted it with distilled water up to 250ml. Wavelength used = 357.9nm, Flame = air-acetylene Following calculations were performed,

Original = 2mL HCl + few drops of nitric acid /0.5g of BaCrO₄

Dilution = 2mL HC1 + few drops of nitric acid /0.5g of BaCrO₄ / 250 ml H₂O.

Cr (absorbance reading) = 7.50 ppm

 $7.80 \times 100 \times 250 \times 100 \square 0.5 \times 10^6 = 19.5\%$

CONCLUSIONS

Following conclusions may be drawn from the experimental work regarding the properties of BaCrO₄. The optimum percentage yield of BaCrO₄ can be obtained from 0.3 M concentrations of sodium chromate and barium chloride at pH 7, temperature 25°C without stirring. The maximum %age yield can be obtained at low temperature e.g. At 25°C the %age yield of BaCrO₄ was 93% and at high temperature as 80°C the yield was minimum i.e. 60%. By increasing the stirring speed the % age yield decreases e.g. when the stirring speed was 20 rpm the yield was 86% and when stirring speed was 60 Rpm the yield was 73%. A maximum %age yield was in the neutral media. The yield was increased in acidic media but decreased in basic media. BaCrO₄ was insoluble in water and acetic acid but soluble in acids and mineral acids.

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