

## STABILITY OF BLEACHING POWDER

M.A. SHEIKH

*Pakistan Belting Mfg. Co. Ltd., Muridke, Pakistan*

(Received June 13, 1973)

**Abstract.** Stable bleaching powder of international standard was manufactured by chlorination of properly slaked lime. Chlorine in 3–5% excess of calculated amount and slaked lime with 1–2% free moisture gave the standard product. The processing difficulties experienced were clogging of the hydration unit and ball formation in the chlorinator. Their causes were investigated. The effect of impurities in lime, temperature and moisture had been discussed. The shelf-life of stable bleaching powder had been studied.

Stable bleaching powder is manufactured from quick lime by slaking and chlorination. Lot of difficulties were observed, e.g. low available chlorine in the finished product, ball formation in the chlorinator during chlorination and the end-product was unstable. It was found experimentally that properly slaked lime containing 1–2% free moisture when chlorinated with a mixture of chlorine and air, gave the product having about 30–36% available chlorine of which 28–33% was stable available chlorine. The chlorine passed was 3–5% in excess of theoretical quantity.

**Experimental**

All these experiments were carried out in a plant having capacity of 10 tons/day. It consisted of a lime hydration unit, rotary chlorinators and drying unit. The presence of free moisture in slaked lime and bleaching powder was estimated by carbide and Dean Stark methods.<sup>1,2</sup> Available chlorine and stable available chlorine were estimated by Bunsen and Wagner's method and according to American Federal Specifications.<sup>3,4</sup>

**Material**

Quick lime used was obtained from indigenous lime stone with the average results given in Table 1.

The quality of lime was good, except calcium carbonate which was present in considerable amount. It was reduced to 5–6% by wind separator after slaking. Carbon and magnesium which affected the stability were well within the limits mentioned by Denora.<sup>7</sup> Dry liquid chlorine, compressed-air and well water were used.

**Results and Discussion**

**Slaking.** Slaking is the most critical step in the manufacture of bleaching powder. The lime and water were appropriately proportioned as to allow for water evaporated as steam due to heat of hydration. Literature revealed that the completely slaked lime with small amount of free moisture is best for chlorination.<sup>5</sup> It had been reported that the slaked lime with 5% free moisture could clog the system.<sup>6</sup>

Practical observations showed that the slaked lime containing up to 10% moisture was free flowing. The reason for clogging of the hydration unit could be explained on the facts that the slaking was exothermic and an induction period 2–3 min was required. The slaking was not completed in the hydrator. The unslaked lime passed into the homogeniser, where the steam liberated during hydration could not escape, unlike hydrator where moisture can pass out through the chimney. The change in angles of the blades of the hydrator allows more time for moisture to escape.

**Chlorination.** According to stoichiometric equation one mole calcium hydroxide reacts with one mole chlorine. Experiments were carried out keeping the feed of calcium hydroxide and air constant and varying the amount of chlorine. The results are given in Table 2.

The rate of flow of chlorine between 60–100 litre per min gave good results.

**Ball Formation.** Two types of ball formation had been observed in the chlorinator, one type consisted of lumps with same structure throughout. The other

TABLE 1. AVERAGE ANALYSIS OF QUICK LIME.

Constituents	Average results obtained (%)
Calcium oxide	72–75
Magnesium oxide	1.0–1.5
Calcium carbonate	20–25
R <sub>2</sub> O <sub>3</sub>	0.1–0.2
Silica	2.5–3.0
Carbon	Not detected

TABLE 2. EFFECT OF CHLORINE ON CALCIUM HYDROXIDE.

Feed of Ca(OH) <sub>2</sub> (g/min)	Flow rate (litre/min)		Available chlorine (%)	Stable available chlorine (%)
	Air	Chlorine		
420	210	140	32.5	29.8
420	210	100	33.7	33.9
420	210	80	33.5	30.7
420	210	60	35.0	34.5
420	210	50	21.9	20.5

type had a fused core. These samples were tested and the results are given in Table 3.

From the above results it was concluded that the lumps which were due to high moisture content, caused the agglomeration of the bleaching powder. These lumps were not harmful and were powdered by increasing the flow rate of air. Ball formation with inner fused mass was due to the presence of unslaked lime. As the chlorination was exothermic, the water liberated during this process reacted with unslaked lime, which was again exothermic, the amount of heat produced was enough to convert bleaching powder into chlorate which further decomposed into chloride. The presence of chloride in fused core was confirmed experimentally.

*Moisture.* Presence of moisture in slaked lime and its effect on chlorination was studied and the results are given in Table 4.

From these results it was concluded that slaked lime with 1-2% moisture gave stable product. Whereas a mixture of slaked and unslaked lime gave unstable product, with low available chlorine. Although it was reported<sup>5</sup> that stable bleaching powder for tropical countries could be obtained by mixing

TABLE 3. ANALYSIS OF BLEACHING POWDER AND BALLS.

Tests	Bleaching powder		
	Powder	Balls	
		Lumps	Fused core
Colour and appearance	Off white	Off white	Off white from outside and light brown from inside
Available chlorine(%)	34.9	36.6	1.5
Stable available chlorine (%)	32.6	33.6	Minimal

TABLE 4. CHLORINATION OF CALCIUM HYDROXIDE.

Feed box analysis (%)				Available chlorine (%)	Stable available chlorine(%)
Ca(OH) <sub>2</sub>	CaO	CaCO <sub>3</sub>	H <sub>2</sub> O		
89.6	—	6.1	2.1	33.7	33.9
91.0	—	6.0	0.6	30.8	29.9
90.6	—	5.4	1.2	35.8	33.2
87.6	2.0	5.8	—	25.0	10.5
85.0	5.0	5.5	—	15.0	5.5
90.0	—	5.0	7.2	29.3	23.9

TABLE 5. EFFECT OF MOISTURE ON BLEACHING POWDER.

Moisture in bleaching powder (%)	Available chlorine (%)	Stable available chlorine (%)
2.3	30.4	28.9
3.5	31.8	30.8
4.4	30.0	23.8
7.2	29.2	23.9
10.5	28.4	21.5
16.3	26.3	20.3

1-2% quick lime with fairly dry bleaching powder just after chlorination. Experiments were carried out but not much improvement in the stability was observed.

Effect of moisture on bleaching powder was studied. The results are given in Table 5.

From the above results it was found that up to 3-5% moisture in bleaching powder, the rate of loss of active chlorine was well within the American Federal Specifications.<sup>4</sup> Bleaching powder is not a stable compound. Removal of moisture was a delicate operation and was handled in the chlorinator rather than in the drying unit.

*Temperature.* The temperature of the chlorinator and the end-product played an important role in determining the quality of the bleaching powder. The temperature of the bleaching powder leaving the chlorinator should be less than 35°C and the

TABLE 6. COOLING OF BLEACHING POWDER.

Available chlorine (%)	Stable available chlorine (%)	Cooled in air		Cooled in closed container	
		Available chlorine (%)	Stable available chlorine (%)	Available chlorine (%)	Stable available chlorine (%)
30.8	29.7	21.6	14.8	30.4	29.4
35.8	34.6	24.0	16.8	34.9	34.1

TABLE 7. COMPARATIVE ANALYSIS OF BLEACHING POWDER.

Tests carried out	Bleaching powder		
	Present work	Imported	Competative local product
Appearance	Off white fine powder	Off white fine powder with considerably amount of fused lumps	Off white fine powder with small amount of dry lumps
Available chlorine (%)	30.8	34.9	28.0
Stable available chlorine(%)	29.5	32.7	24.1
Total chlorine(%)	31.0	38.6	33.0
Calcium carbonate(%)	6.0	4.5	12.0
Moisture (%)	2.3	3.5	6.8

TABLE 8. SHELF-LIFE OF BLEACHING POWDER.

Sample number	Available chlorine (%)	Stable available chlorine (%)	Loss (%)	Moisture (%)	Days
1	32.1	30.5	5.0	1.5	0
2	32.1	30.3	5.6	1.75	20
3	31.8	30.0	5.6	1.8	40
4	31.0	29.0	6.4	2.0	60
5	30.2	28.2	6.6	2.5	80
6	29.9	27.7	7.3	2.7	100
7	29.6	27.0	8.7	3.0	120
8	29.7	27.9	6.0	2.8	140
9	29.9	26.7	10.7	3.0	160
10	29.0	26.8	7.5	2.5	180

temperature difference in the last section of the chlorinator should be about 8.10°C. This was adjusted by controlling the rate of flow of air. Moisture on contact with hot powder decomposed it.

Experiments were carried out to find the best procedure for cooling of bleaching powder to room temperature. The results of such experiments are given in Table 6.

Results from Table 6 revealed that the cooling of bleaching powder out of contact of atmospheric moisture was very important. Comparative studies of bleaching powder from various manufacturers was made and the results are given in Table 7.

The product obtained by the present work was reasonably good.

*Shelf-Life.* Experiments were carried out by keeping samples of bleaching powder from same batch in ten air-tight polyethylene bottles. These were tested one by one within six months. The results are given in Table 8.

From the above results it was found that the maximum loss in available chlorine in 180 days was about 10% and the loss in stable available chlorine in the same period was about 12%. The available chlorine, stable available chlorine and moisture showed no drastic change within six months. These facts indicate that the bleaching powder obtained from properly slaked lime containing small amount of free

moisture and chlorinated with correct amount of chlorine and air gives the product which is reasonably good and stays stable for six months if it is packed properly.

#### References

1. W. Boller, *Chem. Ztg.*, **50**, 537 (1926); R.W. Roberts and A. Fraser, *J. Soc. Chem. Ind.*, **29**, 1977(1910); W.J. Delmhorst., U.S. Patent 2,359,841., (1944).
2. W. Dean and D. D. Stark, *Ind. Eng. Chem.*, **12**, 486(1920).
3. W. Wilfred, *Standard Methods of Chemical Analysis*, (A. I. Vogel,) third edition, p. 363. *Quantitative Inorganic Analysis* (Longman Green, London, 1961), p. 363.
4. American Federal Specifications 'O-C-114a, April 23, 1957.
5. K.E. Raymond and O.F. Donald, *Encyclopaedia of Chemical Technology*, vol. 3, p. 689 (1954); O. Nydegger, *Chem. Met. Eng.* **29**, 1141 (1923).
6. Private communication, Plant Manager, United Chemicals, Kala-Shah Kaku.
7. O. DeNora, *Bleaching Powder Manual* (Impianti Electrochimici, Milano, Italy, 1960)