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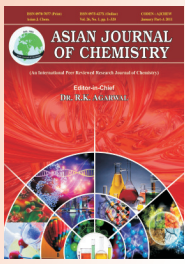

Purification of Byproduct Sodium Chloride from Dibenzoyl Methane Process by Alkali Washing†

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DBD process produces large amounts of by-products of industrial salt. The byproduct is adhered on many kinds of organic compounds and accompanied by bad smell to pollute environment, meanwhile, the useful resource of sodium chloride is wasted. This study adopts alkaline solution to wash the by-product salt and reaches the aim of purification. It is proved the method of alkali solution washing can purify the byproduct of industrial salt effectively in this experiment. With the alkali solution washing, the organics which attached on the surface of salt react with NaOH and change into block floating in liquid at room temperature. The filtrate can be used as washing solution and recycled. The purified salt in the cleaning solution attains the national standard of industrial salt. The salt product is white and odorless. This technology of alkali solution washing to purify industrial salt not only saves the energy compared with the calcination process, but does not produce wastewater as well. By the investigation the optimum technology parameter to wash 100g raw salt is that the pH value of solution and the quality of NaOH are 7-8 and 5-6 g, respectively. The reaction time is 20 min and the residual rate of organics in the salt products is less than 0.3 %.

Keywords: Dibenzoyl methane, Sodium chloride, Alkalinity, Purification.

INTRODUCTION

Dibenzoyl methane (DBM) is an important chemical additive. Its synthetic process uses the methyl benzoate and acetophenone as main materials, sodium ethoxide as catalyst, xylene as solvent[1,2]. Sodium chloride is byproduct during the DBD production. Many kinds of organic materials, such as xylene, DBM and benzoic acid, have been absorbed on the surface of the sodium chloride particles, so the byproduct presents as pale yellow, with a strong pungent odor, pH value is 1-2. The byproduct not only pollutes the environment, but also wastes resources. Therefore, it is necessary to purify the by-product of sodium chloride from the DBM production process. The traditional purification method is calcination[3], the by-product of sodium chloride is put into the furnace and calcined at high temperature. The organic matters on the surface of the particle will be oxidized to carbon dioxide and water, then the byproduct after calcination dissolves in water and deposits, the cleaning solution of NaCl through the process of filtration and evaporation and crystallization to obtain the purified NaCl. But this technology consumes a lot of energy and produces the exhaust gas during calcination, meanwhile, it needs a plenty of water and produces secondary wastewater easily.

In this paper, the method of alkali washing has been investigated to purify the byproduct of sodium chloride. The material is washed in the alkali solution, such as aqueous of NaOH, the organic takes reaction with NaOH and produces insoluble matter floating on the top surface of liquid, then the organic matter is separated from the particle surface and realizes the aim of purification. The alkali solution can be recycled. This method not only improves the purification efficiency and does not produce wastewater.

EXPERIMENTAL

Byproduct of sodium chloride (Bengbu Jiaxian Chemical Lt. Co.), the moisture and organic content in this raw material of industrial salt are around 24 and 4 % (on a dry basis), respectively. Sodium hydroxide (AR), de-ionized water, pH paper, filters, oven, muffle furnace, *etc*.

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Experimental process: Alkali solution was made of sodium hydroxide and de-ionized water. 100 g sample was added into 60 mL of saturated sodium chloride solution, stirring and stillness stratification. The prepared NaOH solution was added into the salt solution drop-by drop, until the pH value of mixture was 6-7. The new product of red organic block was floated in solution and separated by filtration. The filtrate of saturated aqueous NaCl was recycled to wash salt. By the same way it was washed three times until the salt solution without colour and odor. The filtrate was cleaned with saturated brine and vaporized to obtain the purified salt particles.

The purified salt was put into the oven to dry at 80 ºC for 4 h and ensure the moisture in the sample was evaporated entirely, the leavings weight was m_1. The purified and dried sample was put into the muffle furnace at 640 ºC to calcine for 4 h and ensure the residual organic matter was completely oxidized and removed, then the leavings weight was recorded as m_2. The organic residues rate in the salt was calculated by the formula as: $\frac{(m_1 - m_2)}{m_1} \times 100\%$. The lower the rate, the higher purity of salt.

RESULTS AND DISCUSSION

Effect of alkali concentration on organic residual rate: The sodium hydroxide which quality was 0, 1, 2, 3, 4, 5, 6, 7, 8 and 9 g, respectively, was added into 60 mL de-ionized water, to prepare the alkali solution. The 100 g raw salt was washed by solution of different concentrations. Each sample was done three times and measured the average of m_1 and m_2 to calculate the organic residual rate. The experimental result is shown in Fig. 1.

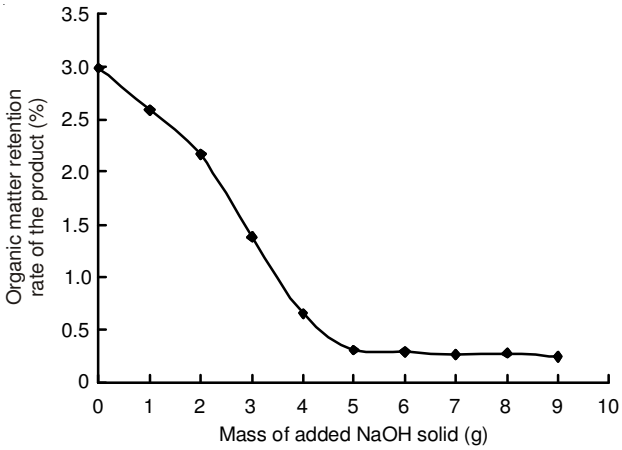

Fig. 1. Relation between alkali quality and the organic residual rate

Fig. 1 shows that organic residual rate decreased with the increasing of NaOH quality. When the quality of NaOH is higher than 5-6 g, the organic residual rate tends to lower and keeps stable. So this value is the optimum point.

According to the national standard of industrial salt[4] (GB/T5462-2003), when the content of effective component of sodium chloride is higher than 98.5 %, the industrial salt is able to used as a refined salt. In the experiment, without calcium and magnesium ions in the raw, the content of sodium chloride is higher than 99 %, so the byproduct of industrial salt can be purified by this method.

Effect of pH value on organic residual rate: The pH value of washing solution changes with the concentration of alkali solution. The initial pH value of raw salt solution was 2.1. The pH value of washing liquid is 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11, respectively in the experiment. Each sample was washed three times to measure and calculate the organic residual rate. The relationship curve shows in Fig. 2.

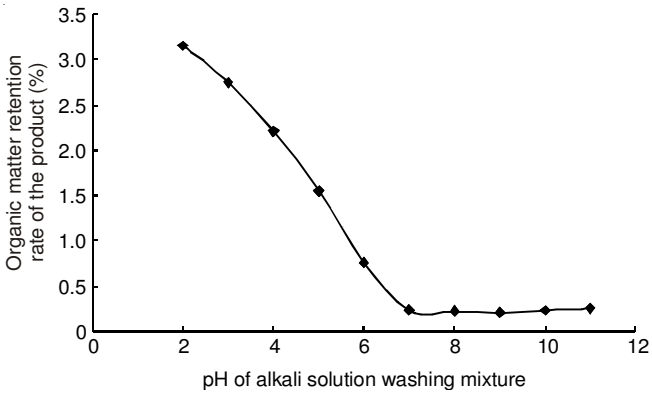

Fig. 2. Relation between pH value and organic residual rate

The organic residual rate decreases with the increasing of pH value in the Fig. 2. When the pH value is higher than 7.2, the organic residual rate tends to stable basically and the minimum value is 0.3 %. Therefore the optimum pH value of washing solution is 7-8.

Effect of washing time on organic residual rate: 5 g NaOH was added into the mixed solution and stirred continuously to wash the byproduct. The washing time is 2, 5, 10, 20, 30 min, respectively. Each sample was done three times. The experimental result shown in Fig. 3.

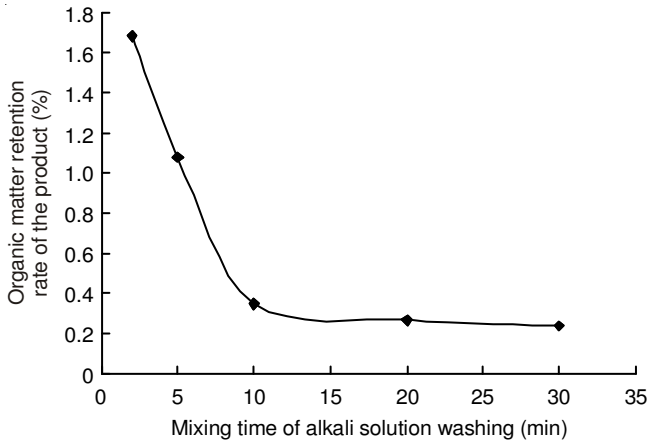

Fig. 3. Relation between washing time and organic residual rate

Fig. 3 shows that when the washing time is less than 5 min, the organic residual rate is 1 %. When the washing time is more than 10 min, the organic residual rate reaches stable and minimum value is 0.27 % basically. Considered the industrial production costs, the optimum washing time is chose as 20 min.

Conclusion

By this experiment it is proved the method of alkali solution washing can purify the byproduct of industrial salt effectively. With the alkali solution washing, the organics which was attached on the surface of salt can react with the NaOH and then change into block floating in liquid at room temperature. The filtrate can be recycled and used as washing solution. The purified salt has been obtained in the cleaning solution and attained the national standard of industrial salt. The salt product is white and odorless. This technology of alkali solution washing to purify industrial salt not only saves the energy compared with the calcination process, but does not produce wastewater as well.

By investigation, the optimum technology parameter to wash 100 g raw salt is that the pH value of solution and the quality of NaOH is 7-8 and 5-6 g, respectively. The reaction time is 20 min and the organics residual rate in the salt products is less than 0.3 %.

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