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Effects of different reaction conditions on the synthesis of 2-hydroxy-4-n-octaoxybenzophenones (UV-531)

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ABSTRACT

2, 4 - dihydroxybenzophenone (UV-214) reacted with 1 - bromododecane in KOH solution to produce 2-hydroxy-4-n-octaoxybenzophenones (UV-531). Effects of different reaction conditions such as the amount of catalyst, the amount of water and the reaction time on the synthesis of 2-hydroxy-4-n-octaoxybenzophenones have been discussed. The optimum molar ratio of 2, 4 - dihydroxybenzophenone to KOH to 1 - bromododecane to dimethyl cetyl ammonium chloride to water and the reaction time were 0.06:0.07:0.06:0.002:0.25 and 6 hours, respectively. The experimental results showed that the yield of 2-hydroxy-4-n-octaoxybenzophenones by adding dimethyl cetyl ammonium chloride into the reaction mixture reached 93.2 %. It was found that dimethyl cetyl ammonium chloride was an ideal phase transfer agent.

KEYWORDS

Reaction condition;
UV-531;
Amount of catalyst;
Amount of water;
Reaction time;
2, 4 - dihydroxy benzophenone (UV-214).

INTRODUCTION

The requirement of polymer aids is increased with the development of three synthetic material industries. 2-hydroxy-4-n-octaoxybenzophenones (UV - 531) and its derivative as UV absorber are one of additive of high polymer materials. 2-hydroxy-4-n-octaoxybenzophenones is used in polyethylene (PE), polystyrene (PS), epoxy resin, unsaturated polyester, coating, synthetic rubber[1]. The traditional synthesis methods of 2-hydroxy-4-n-octaoxybenzophenones are listed as follows: (1) 2-hydroxy-4-n-octaoxybenzophenones is got by using 2, 4 - dihydroxybenzophenone and 1 - bromododecane as feedstocks and KOH as the catalyst. The purity of 2-hydroxy-4-n-octaoxybenzophenones is very high, but the capital of 2, 4 - dihydroxybenzophenone and 1 - bromododecane is very high and the yield of 2-hydroxy-4-n-octaoxybenzophenones is very low. KOH must be added equivalent to that of 2-hydroxy-4-n-octaoxybenzophenones[2]. KOH has to be taken off from this reaction system after the reaction is done. (2) 2, 4 - dihydroxybenzophenone reacts with 1 - bromododecane under the condition of the given temperature and the selected solvent. The cost of feedstock is cheap and easily gets and the yield of the product is high but the purity of the product is very low. A lot of researchers mainly study on how to obtain the higher purity of the product by using activated carbon and vacuum distillation. In this paper, effects of different reaction conditions on the synthesis of 2-hydroxy-4-n-octaoxybenzophenones were discussed.

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EXPERIMENTAL

Feedstock and surfactant

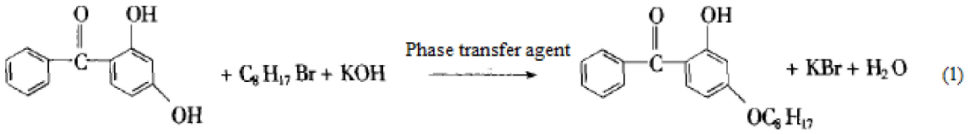
2, 4 - dihydroxybenzophenone (analytical pure, Wuhan Auxiliary Factory); 1 - bromododecane (analytical pure, Shouduang Ocean Chemical Plant); KOH (82%, Shengyang Agent Factory); alkyltrimethylammonium chloride (analytical pure, Dandong Light Chemical Institute) and dimethyl cetyl ammonium chloride (analytical pure, Guangzhou Auxiliary Chemical Plant).

The synthetic method

12.84 g of 2, 4 - dihydroxybenzophenone, 11.58 g of 1 - bromododecane, 4.5 g of distillated water, 4.10 g of KOH and 0.72 g of alkyltrimethylammonium chloride or dimethyl cetyl ammonium chloride were put into 100 ml conical beaker which was heated to 110 °C with stirring. The bath's temperature was kept at between 110 °C and 120 °C. And it continued to stir the conical beaker about 6 hours. The product was cooled, filtered, washed to neutral and dried 40 hours at 90 °C. The melting point of the product measured with the capillary melt point measurement contrasted with that of the analytical pure of 2-hydroxy-4-n-octaoxybenzophenones (the melting point is between 47°C and 49 °C).

The reaction mechanism

2, 4 - dihydroxybenzophenone in KOH solution reacted with 1 - bromododecane to produce 2-hydroxy-4-n-octaoxybenzophenones. The reaction Eq. (1) was listed as follows:



RESULT DISCUSSION

Effects of different amount of catalysts on yields of 2-hydroxy-4-n-octaoxybenzophenones

1 - bromododecane was maldistribution in the distillated water due to its immiscibility. The probability of touching of 2, 4 - dihydroxybenzophenone in KOH solution increased with 1 - bromododecane by adding different catalysts. Effects of different amount of catalysts on yields of 2-hydroxy-4-n-octaoxybenzophenones were shown in TABLES 1 and 2. KOH first reacted with UV-214 to produce anion $C_{13}H_9O_3^-$ which dissolved in the water phase. 1 - bromododecane dissolved in the organic phase. 1 - bromododecane quickly touched and reacted with by increasing the amount of dimethyl cetyl ammonium chloride. The yield of 2-hydroxy-4-n-octaoxybenzophenones increased with the increase of the amount of catalysts (alkyltrimethylammonium chloride or dimethyl cetyl ammonium chloride). Hyamine has two ends. One end has hydrophilic group and another end has lipophilic group. Hyamine can take in KOH solution into organic phase (1 - bromododecane). The length of chain of lipophilic group has an obvious effect on the yield of 2-hydroxy-4-n-octaoxybenzophenones. The longer is the chain of lipophilic group and the more active is the catalytic property. The experimental results showed that the catalytic ability of dimethyl cetyl ammonium chloride was better than that of alkyltrimethylammonium chloride.

TABLE 1 : Effects of different amount of dimethyl cetyl ammonium chloride on yields of 2-hydroxy-4-n-octaoxybenzophenones

Amount of dimethyl cetyl ammonium chloride, g	Amount of UV-531, g	Yield of UV-531, %
0.50	17.55	89.7
0.72	18.23	93.2
0.92	18.30	93.6
1.10	18.41	94.1

Note: the amount of UV-214, 1 - bromododecane, KOH, water and the reaction time were 12.84 g, 11.58 g, 4.10 g, 4.50 g and 6 hours, respectively.

Effects of different amount of water on yields of 2-hydroxy-4-n-octaoxybenzophenones

TABLE 3 presented effects of different amount of water on yields of 2-hydroxy-4-n-octaoxybenzophenones. KOH first reacted with UV-

214 to produce anion which dissolved in the water phase. When the amount of water increased, the alkalinity of decreased. The experimental results showed that the yield of UV-531 decreased with the increase of the amount of water.

TABLE 2 : Effects of different amount of alkyltrimethylammonium chloride on yields of 2-hydroxy-4-n-octaoxybenzophenones

Amount of Alkyltrimethylammonium chloride, g	Amount of UV-531, g	Yield of UV-531, %
0.50	13.38	68.4
0.72	13.71	70.1
0.92	13.97	71.4
1.10	14.18	72.5

Note: the amount of UV-214, 1 - bromododecane, KOH, water and the reaction time were 12.84 g, 11.58 g, 4.10 g, 4.50 g and 6 hours, respectively.

TABLE 3 : Effects of different amount of water on yields of 2-hydroxy-4-n-octaoxybenzophenones

Amount of water, g	Amount of UV-531, g	Yield of UV-531, %
4.5	18.23	93.2
7.0	17.82	91.1
9.0	17.49	89.4
11.0	17.12	87.5

Note: the amount of UV-214, 1 - bromododecane, KOH, dimethyl cetyl ammonium chloride and the reaction time were 12.84 g, 11.58 g, 4.10 g, 0.72 g and 6 hours, respectively.

Effects of different reaction time on yields of 2-hydroxy-4-n-octaoxybenzophenones

TABLE 4 showed effects of different reaction time on yields of 2-hydroxy-4-n-octaoxybenzophenones.

TABLE 4 : Effects of different reaction time on yields of 2-hydroxy-4-n-octaoxybenzophenones

Reaction time, h	Amount of UV-531, g	Yield of UV-531, %
5.0	17.74	90.7
6.0	18.23	93.2
7.0	18.27	93.4
8.0	18.31	93.6

Note: the amount of UV-214, 1 - bromododecane, KOH, dimethyl cetyl ammonium chloride and water were 12.84 g, 11.58 g, 4.10 g, 0.72 g and 4.50 g, respectively.

The yield of 2-hydroxy-4-n-octaoxybenzophenones increased with the increase of the reaction time. The yield of 2-hydroxy-4-n-octaoxybenzophenones almost did not change when the reaction time was more than 6 hours.

CONCLUSION

Effects of different reaction conditions such as the amount of catalyst, the amount of water and the reaction time on the synthesis of 2-hydroxy-4-n-octaoxybenzophenones were studied, and these are summarized as follows:

1) The catalytic ability of dimethyl cetyl ammonium chloride was better than that of alkyltrimethylammonium chloride.
2) The yield of UV-531 decreased with the increase of the amount of water.
3) The yield of 2-hydroxy-4-n-octaoxybenzophenones almost did not change when the reaction time was more than 6 hours.

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