Research Article
Preparation and Application of Stearyl Sodium Lactate

Yang Xuexin

School of Tourism and Cuisine, Harbin University of Commerce, Harbin 150076, China

Abstract: Domestic production of L-lactic acid is used as raw materials for the preparation of a new anionic emulsifier stearoyllactylate, in an index of the acid value and esters value of emulsifiers. It was studied that the effects of temperature, molar ratio, time and catalyst of four factors on the preparation process stearoyllactylate, with the orthogonal experiment method to determine the optimum conditions. Stearoyllactylate structures were identified by IR and the emulsifying properties of stearoyllactate were detected, further studied the effect of stearoyllactylate on dough rheological characteristics and physical properties of bread. Results of the study show the optimal conditions of stearoyllactate synthesis: temperature 105°C, the molar ratio of L-lactic acid and stearic acid is 2.0, the amount of stearic acid catalyst is added to account for 1.5%, the reaction time was 5 h. Such sodium stearoyllactylate was received which emulsifying capacity of 34.85% and solubility of 48.75%.

Keywords: Emulsifier, L-lactic acid, preparation, sodium stearoyllactylate

INTRODUCTION

Stearoyllactylate as excellent food emulsifiers, which in flour products in an amount second only to glycerylmonostearate, ranking second. World annual demand growth rate of 6 to 8%. Stearoyllactylate and calcium current total production capacity of China is only 2,000 tons/year, which is about 1/50 of the United States.

In recent years, domestic synthetic methods on food emulsifiers are mainly stearoyllactylate "two-step" and "three-step method"(Hao *et al*., 2003). The "two-step" the existence of the problem difficult to control the polymerization of lactic acid, which makes the product rate is relatively low. "Three-step" device relatively complex and time-consuming, but also did not consider the adverse effects of lactic acid in the D- and DL-lactic acid on the human body.

Using edible L-lactic acid synthesized stearoyllactylate, improve the traditional synthesis, synthesis stearoyllactylate proposed one-step process, through infrared spectroscopy to identify the product structure and synthesis mechanism to study, which will stearoyllactylate emulsifier provides improved process for preparing food emulsifier as a theoretical basis, but also for the practical application of the necessary experimental data.

MATERIALS AND METHODS

Stearoyl lactate synthesis:
Process: Rawmaterial → catalyst → terification neutralization → Left to dry → detection → package → product → Warehousing.

Procedure: In reaction kettle, according to certain proportion into lactic acid, stearic acid and catalyst, stirring, heating temperature rise to about 105°C, until no moisture volatilizes, cool after the reaction and slowly add sodium hydroxide solution, with the solid content of 10% or so of the emulsion product, then through testing, packaging or finished products.

Analysis method:
Determination of L-lactic acid content and purity:
L-lactic acid content determination: by acid-base titration method:
- According to the relationship between mass and volume, the first volume of the number of lactic acid test and then weigh its weight and then by the formula:

  Density = Quality/volume

- Weigh a certain volume of acid solution, titrated with NaOH standard solution. Averaging triplicate.

L-lactic acid purity determination: by high pressure liquid chromatography:
Determination of the acid value and ester value (Jiao, 1999):
Determination of functional properties:
Determination of solids content: Accurately according to the sample 1 g (accurate to 0.0002), at constant weight in advance over the surface of the dish and in 105±2°C constant temperature drying box, take out sample drying to constant weight:

Solid content (%) = ($G_{after\ drying\ weight}$-$G_{watch\ glass\ weight}$)/($G_{sample\ weight}$ -$G_{watch\ glass\ weight}$)

Determination of solubility (Ning, 1998):
Determination of water holding capacity and oil retention of emulsifier: 5.0 g of sample dispersion in 95 mL of water or oil, stir 25°C for 15 min after let stand for 2 h, 5000 r/min, refuse to go to clear liquid, said the quality of the precipitation:

Water holdup (%) = Precipitation quality/Sample quality×100

Determination of emulsifying properties: According to samples from 1.25 g, decentralization and 25 mL water, add 25 mL soya bean salad oil, electric glass homogenate machine mixing 5 min, 2500 r/min, the centrifugal 5 min, measurement and total height of liquid in the centrifuge tube emulsion layer height:

Emulsifying ability (%) = Emulsion layer height/Liquid total height×100

Foaming and foam stability: The amount of protein product was dissolved in 100 mL of distilled water, pH adjusted to a certain value, then 10000 r/min speed homogenizer homogeneous dispersion 2 min, recording is stopped by volume homogeneous foam, while the protein as a control experiment with Los, bubble calculation as follows:

Blister/% = Homogeneous stops bubble volume (mL) /100 (mL) ×100%

Record homogeneous stop 10, 30, 60, 90, 120 min after the bubble volume, used to measure the foam stability, the above calculation.

RESULTS AND DISCUSSION

L-lactic acid content and purity analysis:
L-lactic acid density: With a pipette moves take 5 mL L -lactic acid, scales on the analysis of the essence of quality is 5.4753 g. Repeated three times in the experiment, we get the L-lactic acid density of 1.09476 g/mL.

L-lactic acid purity analysis: Take 5 mL L-lactic acid diluted into 10% solution in the volumetric flask, add 1 drop of phenolphthalein indicator, with the NaOH standard solution titration to the finish line, mL number to write down the consumption of NaOH. Repeat the experiment, three times get L-lactic acid concentration was 61.78%.

L-lactic acid of high pressure liquid chromatographic analysis: As results shown in Fig. 1, Lactic acid was obtained which optical purity of 90% L-lactic acid.

Screening of neutralization with alkali:
- With NaOH and more complete. High concentrations of NaOH solution dubbed dubbed than Na_2CO_3 concentration. To make the water content of NaOH and less easy to dry.
- When using Na_2CO_3 and a lot of bubbles and cannot fully, resulting in a lot of liquid to escape; and with NaOH and when no such phenomenon.
- Were dried Na_2CO_3 and the acid value; it is difficult to achieve the quality requirements of the acid value.

Stearic acid sodium lactate synthesis process analysis:
Effect of the reaction conditions on L-lactic acid and stearic acid esterification:
Effect of molar ratio on esterification: The molar ratio of the various 1.7, 1.8, 1.9, 2.0, 2.1, 2.2,
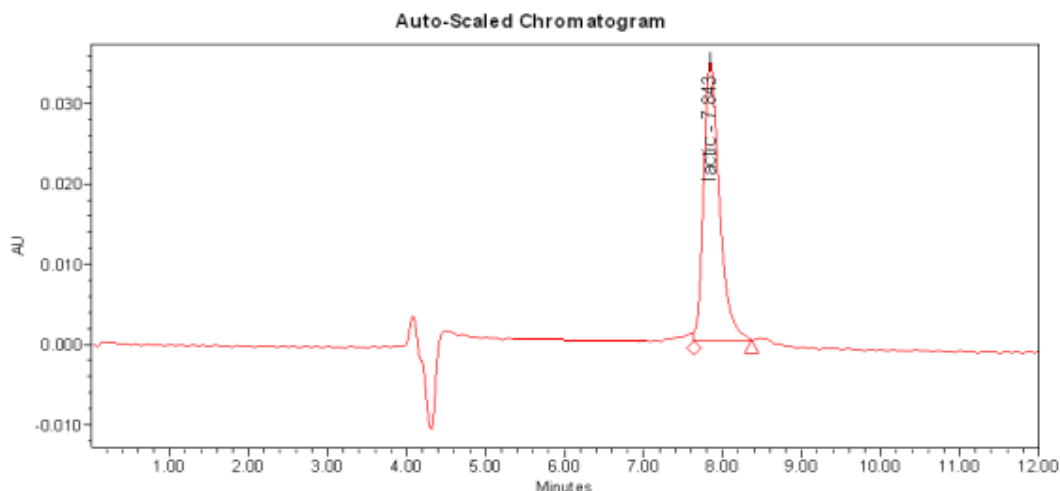

Fig. 1: L-lactic acid liquid chromatogram

respectively at 105°C constant temperature oil bath pot reaction time 4 h, catalyst 1.5% after the reaction and then were measuring their acid value and ester value, choice a better level of molar ratio (Table 1):

- The molar ratio of reaction substance seriously affect the reaction direction of the experiment (Liu *et al.*, 2003). In theory should be 1:1, but lactate itself is a kind of emulsifier, access to lactic acid is not only beneficial to the excessive reaction and in the finished product is lactic acid sodium stearoyl mainly contain a mixture of lactic acid and lactic acid polymer.
- It can be seen from Table 1: in the process parameters for reaction temperature 105°C, 1.5% of catalyst, reaction time, under the condition of 4 h of lactic acid and stearic acid ester value mainly by raw material mole ratio, the influence of the proportion of small, low ester value, along with the increase of the ratio of the ester value also will increase, make it reach to the requirement.

It is better to take 1.9, 2.0, 2.1 level.

Effect of temperature on esterification reaction: The various temperature 85, 95, 105, 115, 125°C respectively, in a molar ratio of 2:1, reaction time 4 h,

1.5% catalyst reaction and then were measured acid value and ester value of their elected Moore good level ratio (Table 2):

- Stearoyl lactate synthesis reaction is endothermic reversible reaction (Chen and Di, 2002), chemical equilibrium theory can be seen from elevated temperature favorable reaction, but the temperature is too high, the color of the finished product becomes too deep dark brown and the acid value will be high, the impact emulsifying properties.
- Experimental data can be seen from Table 2: In the raw material molar ratio of 2.0, the amount of catalyst 1.5%, reaction time 4 h conditions, changing the reaction temperature on the ester value of not obvious, Table 2 data show suitable reaction temperature is about 105°C, the temperature is too high will cause product color deepened.

It is 95, 105, 110°C, respectively to a better level.

Effect of catalyst dosageon esterification reaction: The content of the various catalysts 0.5, 1.0, 1.5, 2.0, 2.5%, respectively at 105°C constant temperature oil bath pot, the molar ratio of 2:1, reaction time 4 H and

Table 1: Effect of different molar ratio on the acid value and ester value of products

Factors	Level					
	1	2	3	4	5	6
Molar ratio	1.7	1.8	1.9	2.0	2.1	2.2
Acid value	66.78	82.35	71.87	74.25	166.03	170.23
Ester value	39.53	81.76	169.87	161.65	179.18	181.25

Table 2: Effect of different temperatures on the acid value and ester value of products

Factors	Level				
	1	2	3	4	5
Temperature/°C	85	95	105	110	115
Acid value	49.90	66.05	74.25	74.63	87.93
Ester value	93.89	103.95	161.65	80.47	81.68

Table 3: Effect of different catalyst dosage on acid value and ester value

Factors	Level				
	1	2	3	4	5
Catalyst /%	0.5	1.0	1.5	2.0	2.5
Acid value	58.03	69.84	74.25	70.69	95.79
Ester value	159.81	126.71	161.65	199.20	214.77

Table 4: Effect of different reaction time on acid value and ester value

Factors	Level						
	1	2	3	4	5	6	7
Time/h	2.0	3.0	3.5	4.0	4.5	5.0	6.0
Acid value	45.60	63.01	62.01	74.25	77.38	158.61	178.16
Ester value	71.97	108.68	66.97	161.65	95.71	124.47	145.35

Table 5: Factors and levels of orthogonal experiment

Levels	Factors			
	A Temperature/°C	B Catalyst/%	C Time/h	D Molar ratio
1	115	1	4	2.0
2	105	1.5	5	1.9
3	95	2.0	3	2.1

Table 6: Orthogonal experiment table

Experiment No.	Factors				Result index	
	A	B	C	D	Acid value	Ester value
1	1	1	1	1	57.09	76.50
2	1	2	2	2	19.62	126.41
3	1	3	3	3	228.09	121.48
4	2	1	2	3	34.82	27.17
5	2	2	3	1	105.62	48.47
6	2	3	1	2	102.98	94.01
7	3	1	3	2	169.21	44.80
8	3	2	1	3	140.53	122.49
9	3	3	2	1	127.05	156.43
Acid value	K_1 304.90	261.12	300.60	289.76	T = 985.01	
					Y = 109.45	
	K_2 243.42	265.77	181.49	291.81		
	K_3 436.79	458.12	502.92	403.44		
	k_1 101.63	87.04	100.20	96.59		
	k_2 81.84	88.59	60.56	97.27		
	k_3 145.60	152.71	167.64	134.48		
	R 64.46	65.67	107.14	37.89		
	Optimal levels A_2	B_1	C_2	D_1		
Ester value	K_1 324.39	148.47	293.00	281.40	T = 817.76	
					Y = 90.86	
	K_2 169.65	297.37	310.01	265.22		
	K_3 323.72	371.96	214.75	271.14		
	k_1 108.13	49.49	97.67	93.80		
	k_2 86.55	99.12	103.34	88.41		
	k_3 107.91	123.97	71.58	90.38		
	R 51.58	74.48	31.76	3.42		
	Optimal levels A_1	B_3	C_2	D_1		

then they were measured acid value and ester value, choose a better level molar ratio (Table 3):

- This reaction is an esterification reaction, the catalyst is more conducive to the reaction (Mattey, 1992). Concentrated sulfuric acid as a catalyst is preferable, such as concentrated sulfuric acid value of the finished product will increase too much.
- Table 3 data show that the reaction conditions are the same in other cases, so they chose catalyst for the quality of stearic acid 1.9, 2.0, 2.1%, which are better levels.

Effect of reaction time on esterification reaction:
Different reaction time 2.0 h, 3.0 3.5 h, 4 h, h, h, h, 6.0 5.0 4.5 h at 105°C, respectively, the constant temperature oil bath pot, molar ratio of 2:1, catalyst reaction, 1.5%, respectively, to determine the acid value and the ester value them, chooses the mole ratio of good level (Table 4):

- According to the information, too long, the acid value of the finished product will be increased (Ling, 2003). We can conclude from data of Table 4, between 5 h to 6 h acid value soared, indicating a very obvious change in between.
- The longer the time, the greater the degree of aging of the catalyst.
  It is a better level of factor of 4, 5, 6.

Test of optimal reaction conditions:
Design of orthogonal experiment: From the above discussion of each variable, based on the results of

single factor experiments, the acid value and ester value index to determine the factors and orthogonal experiment (Table 5).

Orthogonal experiment table: Choose appropriate levels of factors of reaction substance, with $L_9 (3^4)$ orthogonal experiment. According to the orthogonal table $L_9 (3^4)$ experiment (Table 6).

From the data in Table 6, with the measured value of stearoyl lactate and acetate acid number two indicators, with range and variance analysis, optimal synthesis.

By the orthogonal experiment table analysis data and Fig. 2 acid value pictorial diagram you can see, the influence of the factors: the influence of time on the acid value maximum, other factors, the smaller the impact order to C>>A>B>D. Catalyst had the greatest influence on the value of an ester, mole ratio of the minimum, the influence order of C>B>A>>D.

As can be seen in Fig. 3 intuitively optimal level acid value is: $A_2B_1C_2D_1$. The optimal level of ester value is: $A_1B_3C_2D_1$.

Between the two can be seen that the optimal time factor and the molar ratio to reach agreement, the effect of temperature on the acid number of relatively large impact on the value of the ester is not obvious, take A_2. In the catalyst, the changes have to ester value of large, so take B_2.

Taken together, the best synthesis process for lactic acid sodium stearoyl: $A_2B_2C_2D_1$ the reaction conditions of temperature is 105°C, 1.5% content of catalyst, reaction time of 5 h, mole ratio of 2.0.
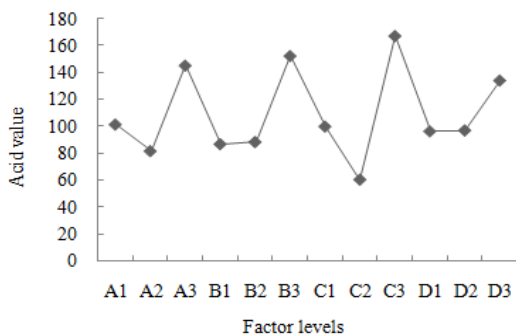
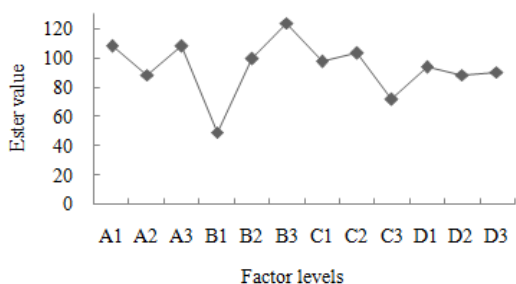Fig. 2: Pictorial diagram of acid value

Fig. 3: Pictorial diagram of ester value

Table 7: Comparison of solid and liquid
	Acid value/mgKOH/g	Ester value/mgKOH/g
Solid	83.48	183.18
Liquid	60.13	103.22

Table 8: Physical and chemical properties of SSL
Emulsifying ability	Solubility	Water holdup	Oil holdup	Solid content
34.85%	48.75%	80.92%	241.67%	25.74%

Table 9: Bubble volume changes
Time/min	10	30	60	90	120
Bubble volume/ml	48	45	44	40	39

Verification experiment and the physical and chemical properties testing of stearoyllactylate:
Verification experiment: Under optimum conditions: the temperature of the reaction conditions of 105°C, catalyst content of 1.5%, reaction time 5 h, the molar ratio of 2.0. Repeat the above optimum conditions, the measured obtaining an acid value of 83.48 mg/KOH and ester value 183.18 mg/KOH detect physical and chemical indicators.

Physical and chemical properties:
Emulsifying ability: 34.85% stearoyllactylate, monoglycerides of 46.97%.

And after the reaction, divided into two parts and dried into a solid, the other part is not dried into liquid form. They were measured in solid and liquid form acid value and ester value (Table 7).

As can be seen from Table 7, are higher than the acid value of solid and liquid ester value. Solid products were solubility, solids content, emulsification, water holdup, hold oil yield measurements (Table 8).

According to the different solubility, emulsifier can roughly difference between the relative polarity. Smaller polarity hydrophilic emulsifier and hydrophilic substances in water solubility have larger and can promote the formation of the oil-in-water type emulsion (Jiao, 1999). Lactic acid sodium stearoyl is oil-in-water type emulsifier. See from Table 8, the solubility of sodium stearoyl lactic acid was 48.75%, the solubility is relatively large.

Sodium stearoyllactylate foaming: 50%. Measure its foaming stability as shown in Table 9.

Bubble is a thermodynamic instability system, easy to burst, here refers to the foam stability is relatively stable in a period of time (Bera and Mukherjee, 1989). Factors affecting the stability of foam mainly has the following kinds:

- The foam on the surface of the "repair"
- The surface viscosity
- The surface charge

The greater the stability of the rejection of the charge, the greater the bubble (Xu, 2002). Stearic acid sodium lactate is anionic emulsifier, it can be ionized in aqueous solution to form negatively charged ions by organic interface (Lin and Zayas, 1987). So the stability of the bubble can be seen from Table 9 is better, change in 2 h from 48 to 39, fell by about 20%.

CONCLUSION

The results show that stearoyl lactate synthesis of optimal conditions: temperature 105°C, the molar ratio of L-lactic acid and stearic acid is 2.0, the amount of catalyst is added to account for 1.5% of stearic acid, 5 h. The resulting product acid value 83.48 mgKOH/g and an ester value 183.18 mgKOH/g, can reach FAO/WHO (1995) quality requirements. Infrared spectrophotometer identify the molecular structure of the product is esterified L-lactic acid and stearic acid. 34.85% emulsification product solubility was 48.75%. The results showed that sodium stearoyllactylate is a good food additive.

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