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RESEARCH ARTICLE

Features of triamyl citrate synthesis

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Abstract

Objectives. *To find an effective way for obtaining triamyl citrate, an environmentally friendly, biodegradable citric acid ester used as a plasticizer for PVC-based polymer compositions.*
Methods. *The possibilities of heterogeneous catalysis were analyzed using the case study of three commercial samples of macroporous sulfocationites (Amberlyst™ 15, Amberlyst™ 70, and TULSION® 66). Homogeneous catalysis was studied using the example of orthophosphoric acid (H_3PO_4), while self-catalysis was investigated during esterification of citric acid with amyl alcohol (ROH). The syntheses were carried out under identical conditions: T = 110°C, the ratio of CA:ROH = 1:5 (mol) amount of catalyst 1 wt % on the reaction mass in a thermostatically controlled reactor of ideal mixing with continuous distillation of the resulting water.*
Results. *It was found that in all variants (even under self-catalysis conditions), the conversion of citric acid in 180 min reached 94–99%. Triamyl citrate was formed after 9 h with a yield of 90% only when using a homogeneous catalyst (H_3PO_4) and in the presence of a heterogeneous catalyst sample (Amberlyst™ 15).*
Conclusions. *The revealed differences in the reactivity of the studied sulfocationites (Amberlyst™ 15, Amberlyst™ 70, and TULSION® 66) confirm the well-known theoretical positions, according to which the kinetic pseudo-homogeneous model of the esterification process of hydroxy acids in excess of aliphatic alcohols is based on the law of acting masses and depends on the specific surface area of the catalyst, which for Amberlyst™ 15 is of the greatest importance as compared to Amberlyst™ 70 and TULSION® 66 (m^2/g): 53:36:35, respectively.*

Keywords: *citric acid, amyl alcohol, esterification, heterogeneous catalysts, self-analysis*

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НАУЧНАЯ СТАТЬЯ

Особенности синтеза триамилцитрата

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Аннотация

Цели. *Поиск эффективного метода получения триамилцитрата – экологически чистого, биоразлагаемого сложного эфира лимонной кислоты, используемого в качестве пластификатора полимерных композиций на основе поливинилхлорида.*

Методы. *Выявлены возможности гетерогенного катализа на примере трех коммерческих образцов макропористых сульфокатионитов (Амберлист™ 15, Амберлист™ 70 и Тулсион® 66); гомогенного катализа на примере ортофосфорной кислоты (H_3PO_4) и самокатализа при этерификации лимонной кислоты (ЛК) амиловым спиртом (ROH). Синтезы проводили в одинаковых условиях: T = 110 °C отношение ЛК:ROH = 1:5 (мольн.) количество катализатора 1 мас. %. на реакционную массу в термостатированном реакторе идеального смешения с непрерывным отгоном образующейся воды.*

Результаты. *Установлено, что во всех вариантах конверсия лимонной кислоты за 180 мин достигает 94–99%. Триамилцитрат с выходом 90% образуется через 9 ч только при использовании гомогенного катализатора (H_3PO_4) и в присутствии образца гетерогенного катализатора – Амберлист™ 15.*

Выводы. *Выявленные различия в реакционной способности исследованных сульфокатионитов Амберлист™ 15, Амберлист™ 70 и Тулсион® 66 подтверждают известные теоретические положения, в соответствии с которыми кинетическая псевдогомогенная модель процесса этерификации гидроксикислот в избытке алифатических спиртов основывается на законе действующих масс и зависит от удельной поверхности катализатора, которая для Амберлист™ 15 имеет наибольшее значение по сравнению с Амберлист™ 70 и Тулсион® 66 ($м^2/г$): 53:36:35 соответственно.*

Ключевые слова: *лимонная кислота, амиловый спирт, этерификация, гетерогенные катализаторы, самокатализ*

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INTRODUCTION

Citric acid (CA) is widely used in various sectors of the world economy as a common acidity regulator, antioxidant, and complexing agent. With a current global annual production volume of 1.8 mln t/year, the annual increase in demand is projected at the level of 4.0–4.5%.

The structure of citric acid consumption in Russia differs from the world by a much smaller share of its use in industries that form the basis of environmentally

friendly synthetic detergents, solvents, plasticizing compositions: 80% of citric acid is used in the food industry, with 10–15% being allocated to the production of detergents, and up to 10% in cosmetics and pharmaceuticals [1].

The dominant portion of citric acid derivatives is comprised of its esters based on higher aliphatic alcohols. These belong to the Hazard Class 4 considered as non-toxic. Products having a flash point of at least 168°C (according to GOST 8728-88[1]) have plasticizing properties and provide foaming of polyvinyl chloride (PVC) back foam pastes [2, 3].

To date, the production of higher alkyl citrates in Russia has been limited due to the lack of a raw material base. However, within the framework of the general development of gas chemistry and the increase in the capacity of oxosynthesis processes, it is expected to create a sufficient potential of alcohols C_3–C_5 and higher over the next 20–25 years [4]. The product of esterification of citric acid with amyl alcohol is of the greatest interest.

The technological processes of synthesis of esters are divided into two groups [5–7]:

1) liquid-phase group, i.e., thermal in self-catalysis mode or homogeneous-catalytic, in which the chemical reaction is combined with the process of distilling volatile products;

2) heterogeneous-catalytic group, carried out in liquid or gas phases in flow apparatuses without combining with separation processes.

The processes of the first group are traditional and the most common in the technology of esterification. However, they work satisfactorily only at a high rate of chemical reaction, otherwise the completeness of the transformation and the reactor performance are too low.

It is known that the longer the length of the hydrocarbon radical of the aliphatic alcohol, the higher the molecular weight of the target product and the flash point. This reduces the emission of the plasticizer, improves its operational properties [8]. However, it was found that an increase in the hydrocarbon radical on the CH_2-group in the initial alcohol reduces the reactivity of the alcohol by an average of 1.3 times and increases the reaction time [9].

Although the use of homogeneous catalysts in industry (*p*-toluene sulfonic acid, orthophosphoric acid, methanesulfonic acid) in esterification reactions offers a good catalytic effect, it is associated with high production costs and negative ecological effects [5]. Therefore, there is a current trend involving the use of heterogeneous catalysts. This eliminates many disadvantages associated with the use of homogeneous catalysts, since their heterogeneous counterparts are easily separated from the reaction mass by decantation or filtration, offer an effective means of reducing or eliminating corrosion problems, as well as suggesting the possibility of switching from a semi-periodic to a continuous process.

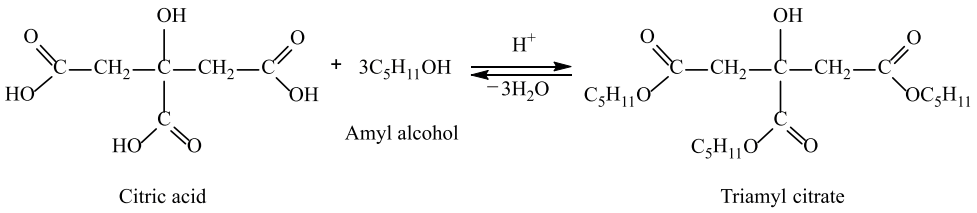
The disadvantages of using heterogeneous catalysts—high temperature, high catalyst consumption, and increased side reactions, lead to tarring of the reaction mass and a decrease in the quality of the target products [10–11].

While the growing interest of domestic producers of citric acid esters is justified by the need to achieve independence from imports, a thorough assessment of the effectiveness of technological solutions and the quality of the target products is required. This position becomes especially significant when expanding the raw material base towards the use of hydroxy acids and high-molecular alcohols of linear and isostructure.

Therefore, as part of this study we conducted comprehensive studies and compared the possibilities of self-catalysis, homogeneous and heterogeneous catalysis during esterification of citric acid with amyl alcohol in order to obtain triamyl citrates with a yield of 85–90%.

EXPERIMENTAL

As initial raw materials, the following substances, which differ in geometric dimensions, were chosen: Citric acid (food grade, monohydrate with a base



Citric acid — Triamyl citrate

Scheme. Esterification of citric acid with amyl alcohol.

[1] GOST 8728-88. Interstate Standard. Plasticizers. Specifications. Moscow: IPK Izd. Standartov; 1988.

substance content of 92.5%) (*Reagent*, Russia); pure grade amyl alcohol with a purity of more than 99% (*Reachim*, Russia); pure grade orthophosphoric acid (homogeneous catalyst) with a purity of 85% (*Reagent*, Russia); three commercial industrial samples of microporous sulfocationites: Amberlyst™ 15 (*Sigma-Aldrich*, Germany), Amberlyst™ 70 (*Sigma-Aldrich*, Germany) and TULSION® 66 (*Thermax*, India).

The characteristics of the catalysts are given in Table 1.

Esterification of citric acid with amyl alcohol was carried out in a thermostated mixing reactor. After dissolving citric acid in amyl alcohol in the reactor, a catalyst was introduced and samples were taken at specified intervals, with the change in the concentration of carboxyl groups in the reaction mass determined by titrimetry. The composition of the reaction mass was determined using gas–liquid chromatography on the Crystal 2000M chromatographic complex (*Chromatec*, Russia) with the following parameters: capillary column with grafted nonpolar phase OV–101 100 m × 0.2 mm × 0.2 μm; column temperature mode: 120°C (10 min)—15°C/min—260°C; evaporator temperature 300°C; detector temperature 300°C; carrier gas is helium, flow division 1/80. In the analysis, a derivatization method was used to convert polar organic compounds containing carboxyl groups into less volatile ones. Diazomethane was used as a derivatizing agent. The internal standard method was used for the quantitative determination of citric acid esters in the reaction mass. Dicyclohexyl adipate was used as a standard [12].

Based on previous works [2–3, 9, 12], in which data on the effect of temperature, molar ratio of reagents, type, and quantity of homogeneous catalysts were obtained, the following research conditions were selected: temperature 110°C; citric acid:alcohol ratio is 1:5 (mol); amount of catalyst is 1% per reaction mass.

All experiments were carried out in the mode of distillation of the released reaction water with strict observance of the isothermal regime.

Table 1. Characteristics of the catalysts used in the study

Acid	Formula of the substance	Dissociation constant K_a	pK_a
1. Self-catalysis			
Citric acid	$HOOCCH_2C(OH)(COOH)CH_2COOH$	$K_1 = 7.41 \cdot 10^{-4}$ $K_2 = 1.74 \cdot 10^{-5}$ $K_3 = 9.80 \cdot 10^{-6}$	3.13 4.76 5.40
2. Homogeneous catalysis			
Orthophosphoric acid	H_3PO_4 (85%)	$K_1 = 7.59 \cdot 10^{-3}$ $K_2 = 6.17 \cdot 10^{-8}$ $K_3 = 4.20 \cdot 10^{-13}$	2.12 7.21 12.38
3. Heterogeneous catalysis			
No.	**Indicators**	**Amberlyst™ 15**	**Amberlyst™ 70**	**TULSION® 66**
1	Minimum capacity, eq/kg	4.7	2.55	5
2	Shipping weight, g/L	610	770	500
3	Specific surface area, m^2/g	53	36	35
4	Pore diameter, Å	300	220	450–500
5	Recommended max. op. temperature, °C	<120	190	130

RESULTS AND DISCUSSION

Figures 1–5 demonstrate graphs of the evolution of products obtained as a result of research for 180 min.

Table 2 summarizes the results of all experimental studies performed in this work in the range of 180–540 min.

Citric acid was found that to be rapidly esterified by the first carboxyl group with the formation of monoamyl citrates in all variants of catalysis (even under self-catalysis conditions), which corresponds to the literature data [6, 13, 14]. In 180 min, the conversion of citric acid reaches 94–99%. Regardless of the type of catalysis, the content of diamyl citrate for 240 min is 50–60%. Esterification of the third carboxyl group into triamyl citrate is limiting; the yield of triamyl citrate in 84–90% is achieved in 9 h (540 min) only with homogeneous catalysis and in the presence of Amberlyst™ 15 catalyst.

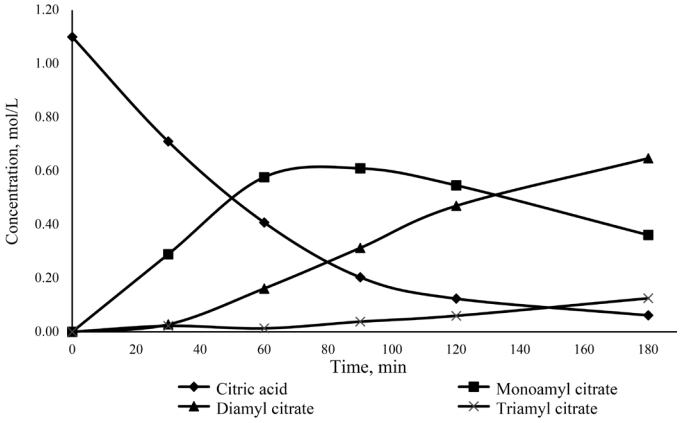


Fig. 1. Esterification of citric acid under conditions of self-catalysis.

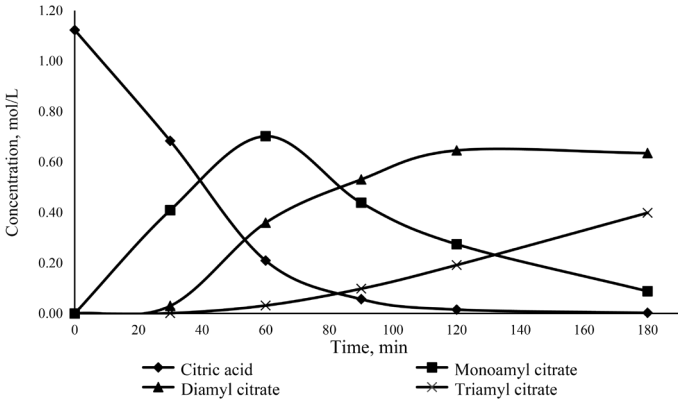


Fig. 2. Esterification of citric acid in the presence of orthophosphoric acid.

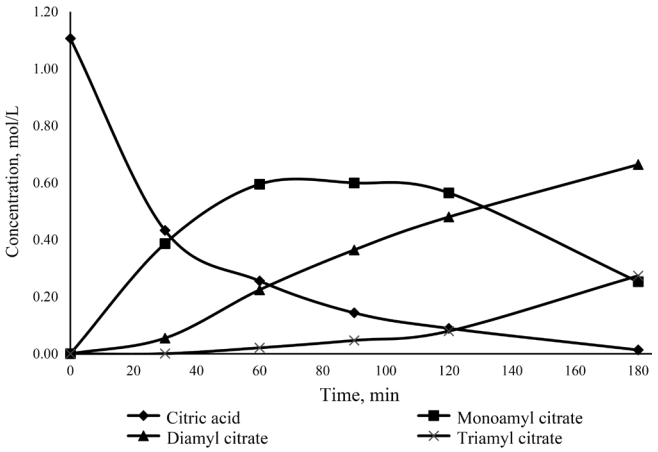


Fig. 3. Esterification of citric acid in the presence of Amberlyst™ 15.

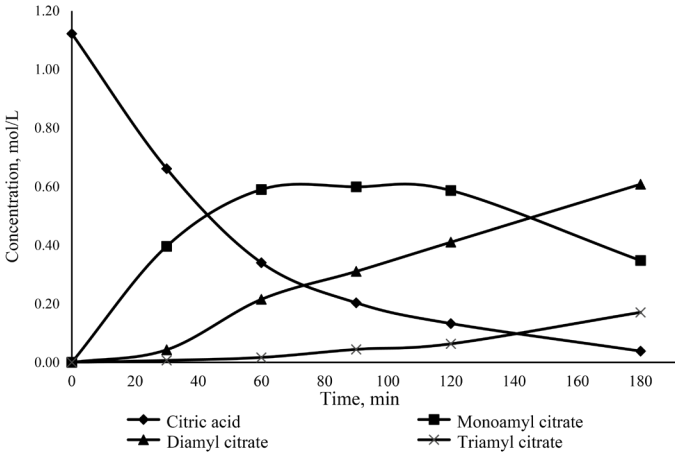


Fig. 4. Esterification of citric acid in the presence of Amberlyst™ 70.

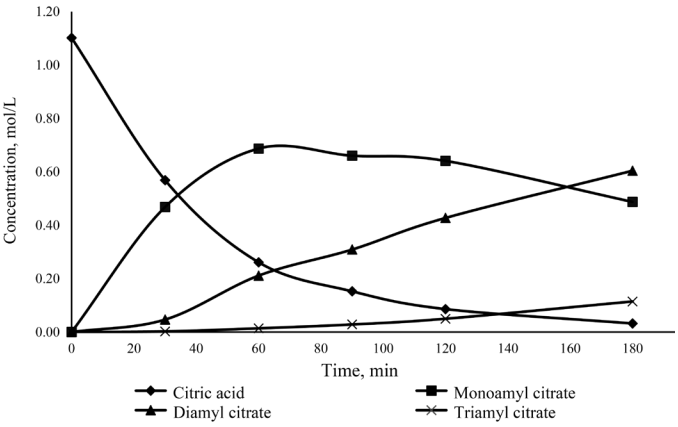


Fig. 5. Esterification of citric acid in the presence of TULSION® 66.

Table 2. Composition of the reaction mass of the esterification reaction of citric acid with amyl alcohol at 110°C, reaction time: 180, 240, and 540 min with the distillation of reaction water

Experiment / Yield, %	Self-catalysis	Amberlyst™ 15	Amberlyst™ 70	TULSION® 66	H_3PO_4
180 min					
Citric acid (CA)	5.13	1.10	3.31	2.57	0.21
Monoamyl citrate	30.23	20.99	29.85	39.39	7.85
Diamyl citrate	54.17	55.16	52.17	48.82	56.48
Triamyl citrate	10.47	22.75	14.67	9.25	35.44
Conversion by CA, %	94.40	98.80	96.50	97.10	99.78
240 min					
CA	1.52	0.31	0.99	0.85	0
Monoamyl citrate	19.09	8.38	14.53	21.99	2.53
Diamyl citrate	58.32	51.55	55.07	56.18	48.20
Triamyl citrate	21.07	39.76	29.46	20.98	49.26
Conversion by CA, %	98.42	99.66	98.97	99.14	100
540 min					
CA	0	0	0	0	0
Monoamyl citrate	0.80	0	0.68	1.07	0
Diamyl citrate	32.54	10.32	30.63	25.35	15.74
Triamyl citrate	66.66	89.68	68.69	73.58	84.26
Conversion by CA, %	100	100	100	100	100

In Russia, about 300000 t/year of citric acid are obtained from the waste of molasses sugar production [15]. From this amount of citric acid, it is possible to obtain about 150000 t/year of citric acid esters comprised of trialkyl citrates, of which the esters of citric acid and amyl alcohol have the greatest practical significance as a PVC plasticizer [16].

CONCLUSIONS

The revealed differences in the reactivity of the studied sulfocationites (Amberlyst™ 15, Amberlyst™ 70, TULSION® 66) confirm the well-known theoretical positions, according to which the kinetic pseudo-homogeneous model of the process of esterification of hydroxides by aliphatic alcohols (in excess) is based on the law of acting masses. In this regard, the reaction rate will depend on the specific surface area of the catalyst, which for Amberlyst™ 15 is of the greatest importance compared to Amberlyst™ 70 and TULSION® 66 (m^2/g): 53:36:55, respectively.

Based on the obtained results, it can be assumed that the installation of semi-continuous azeotropic esterification of citric acid in excess of alcohol using sulfocationite with a large specific surface area as a catalyst will be the most effective.

Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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