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NEW POLYVINYLCHLORIDE PLASTICIZERS

Extended Abstract:

One of the main large-capacity polymers of modern chemical industry is polyvinylchloride (PVC). Polyvinylchloride is characterized by many useful engineering properties – chemical firmness in different environments, good electric properties, etc. It explains immensely various use of materials on the basis of PVC in different engineering industries. It is cable, building, light industries, mechanical engineering and automotive industry where PVC is widely applied. One of the reasons why PVC production is dramatically growing is that there is no yet other polymer which could be subjected to such various modifying as it is done with PVC.

However under normal temperature this polymer is fragile and isn't elastic that limits the field of its application. Rapid growth of production of polyvinylchloride is explained by its ability to modify properties, due to introduction of special additives when processing. Introduction of plasticizers – mostlly esters of organic and inorganic acids – into PVC allows significant changing properties of polymer. Plasticizers facilitate process of receiving polymeric composition, increase flexibility and elasticity of the final polymeric product due to internal modification of polymeric molecule.

This paper presents the results of research on production methods, physico-chemical and mechanical properties of new chemical additives of polyvinylchloride – plasticizers based on oxyalkylated alcohols. It also describes the optimal conditions for the compounds synthesis and the results of experiments performed with these compounds as additives in PVC film compositions. It is noted that composition obtained by introducing developed plasticizers into PVC compositions meet the requirements of the existing standards, and their oil and petrol resistance exceed standard samples.

Key words: oxyalkylated phenols and butanols, phthalates of oxyalkylated alcohols, PCV plasticizers, PVC film, oilresistance, petrolresistance, elongation at break, meltflow index, breaking strength, brittleness temperature, heat stability.

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Polyvinylchloride (PVC) is one of the most demanded large-capacity polymers made in Russia and abroad, following only polyethylene by production volume. The range of materials and products based on it continues to grow, because they meet the high requirements of modern processing and operating conditions [1–4].

Further development of polyvinylchloride production accompanied by continuous extention of scope first of all is determined by development of functional chemicals additives – stabilizers and plasticizers.

Stabilizers are introduced into composition of polymers to decelerate their ageing that caused mainly by destruction. A large number of chemical compounds are used as stabilizers: heat stabilizers reduce harmful effects of thermal and thermooxidizingdegradation of polymer; antiozonants protect polymers from effect of atmospheric oxygen and ozone; light stabilizers slow down aging of polymers under the influence of ultra-violet light; antirads protect polymers from destruction under the influence of high-energy radiations; passivators of polyvalent metals protect polymers from the destroying effect of metal «poisons»; antifatiguesprotect polymeric materials, mainly rubbers, from cracking at action of variable loadings [5–7].

The largest segment of the additives market is the market of plasticizer. Introduction of plasticizer into polymeric composition makes it possible to produce material with specified elasticity for the wide range of temperatures. The proper selection of polymer facilitates the polymer processing, greatly increases the frost resistance, flame resistance and improves many other properties of polymer soft products [8].

Various classes of chemical compounds are studied and used as plasticizers: esters ofterephthalic, aliphatic, dicarboxylic, trimellitic and pyromellitic acids; polyester and phosphoricesters; epoxidizedester of soy, palm oils, of tallow fatty acids and other compounds (for example, higher alcohols from C_{18}, paraffin wax) [9, 10].

In recent years there has been a purposeful development of chemical additives. Thus complex stabilizers containing all necessary components, including lubricants have been already in use. Toxic cadmium, barium, leadcontaining heat stabilizers are replaced by non-toxic carboxylates of alkaline earth metals. New plasticizers, giving to plastic compound specific properties, are developed [11].

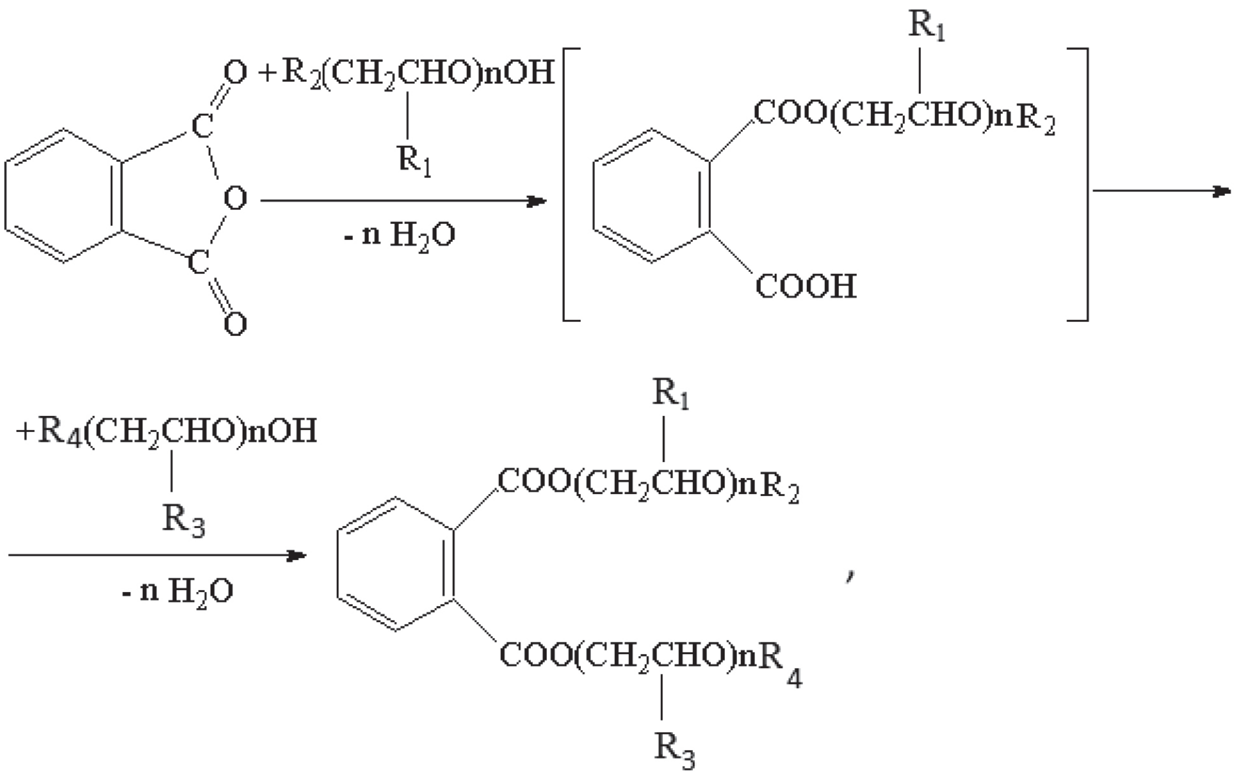
This paper presents the results of research on development of PVC films compositions with new oil and petrol-resistant plasticizers.

Preobtained calcium-zinc complex salts of oleic, stearic and alpha branched saturated monocarboxylic acids (VIC) were used as heat stabilizers [12, 13].

Phthalates of oxyalkylated alcohols (Fig. 1). Initial oxylated and hydroxypropylated butanol and phenol obtained previously were used as plasticizers [14–17].

Conditions for obtaining the desired products are shown in Table 1, physicochemical parameters are given in the Table 2.

Obtained esters were tested as PVC plasticizers in compositions of PVC films.



where $R_1 = H$, $R_2 = C_4H_9$, $R_3 = H$, $R_4 = C_4H_9$ (I); $R_1 = CH_3$, $R_2 = C_4H_9$, $R_3 = CH_3$, $R_4 = C_4H_9$ (II); $R_1 = H$, $R_2 = C_6H_5$, $R_3 = H$, $R_4 = C_6H_5$ (III); $R_1 = CH_3$, $R_2 = C_6H_5$, $R_3 = CH_3$, $R_4 = C_6H_5$ (IV); $R_1 = H$, $R_2 = C_6H_5$, $R_3 = H$, $R_4 = C_4H_9$ (V); $R_1 = CH_3$, $R_2 = C_6H_5$, $R_3 = CH_3$, $R_4 = C_4H_9$ (VI); $R_1 = CH_3$, $R_2 = C_6H_5$, $R_3 = H$, $R_4 = C_4H_9$ (VII); $R_1 = H$, $R_2 = C_6H_5$, $R_3 = CH_3$, $R_4 = C_4H_9$ (VIII).

Fig. 1

Compositions of the films and the experiment result of obtained esters as plasticizers in commercial formulations of PVC films are shown in Table 3 (Formulation (wt parts): PVC – 100; plasticizer – 50; Ca–Zn complex stabilizer – 3.0).

Prepared compositions were milled in laboratory mill 320 PD at the temperature of 160–162°C for 10 minutes. There were no any difficulties during rolling of composition: the film didn't stick to the rolls, holes, chips and cracks were absent in the obtained samples of plastic. Moreover, during the production of PVC compositions in the mixer and their processing on rollers technological difficulties did not arise.

Physical, physicochemical and physico-mechanical properties were determined in accordance with standard methods. The strength and elongation

Table 1

The optimum conditions for obtaining chemical additives

№	Reagent molecularratio		Temperature, °C	Note
1	alcohol : ethylene (propylene) oxide	1:1.3-2.2	110–180	amount of catalyst 0.5–3% (wt. by loading)
2	phthalic anhydride: oxyalkylated alcohol	1:2	121–170	amount of catalyst 0.1–2% (wt. by loading) + activated carbon in an amount of 1% (wt. of weight of the loaded components)

Table 2

Physicochemical parameters of synthesized esters

Indicator name	Plasticizers							
	I	II	III	IV	V	VI	VII	VIII
Oxyalkylation degree, n	2.0	2.2	1.0	2.1	2.0*	2.2*	2.0*	2.2*
Density, d^{20}_{4}	1.4816	1.4745	1.1086	1.1002	1.1081	1.1050	1.1062	1.1060
Index of refraction, n^{20}_{D}	1.0757	1.02991	1.5194	1.4924	1.5183	1.5174	1.5178	1.5178
Acid number, mg KOH/g	0.1	0.4	0.1	0.1	0.2	0,2	0,2	0.2
Ether index, mg KOH/g	243	207	273	198	257	203	211	235
Molecular mass, found	461	541	411	566	436	553	514	476
Freezing temperature, °C	–50	–44	–40	–39	–40	–37	–39	–40
Weight fraction of volatile compound (100°C, 6 h),%	0.25	0.25	0.10	0.15	0.10	0.10	0.12	0.10
Flash-point, °C	200	Higher than 200	200	199	200	200	200	200

* phenol ethoxylation degree = 1.0; phenol hydroxypropylation degree = 2.1

Table 3

Physicochemical parameters of synthesized esters

Indicator name	Test samples							
	I	II	III	IV	V	VI	VII	VIII
Stress at elongation 100%, MPa	11.9	11.8	12.5	12.4	12.1	12.2	12.1	12.2
Breaking stress, MPa	22.6	22.4	23.1	23.0	22.9	23.0	22.8	23.1
Elongation at break, %	286	287	289	289	287	289	288	288
Frost resistance, °C	–34	–34	–37	–39	–34	–34	–36	–37
Mass losses at 130°C, during 6 h., %	2.1	2.2	2.5	2.7	2.8	2.6	2.8	2.7
Extractibility by water, %	0.305	0.301	0.324	0.325	0.319	0.316	0.318	0.316
Water absorption ability, %	0.472	0.469	0.484	0.486	0.474	0.478	0.479	0.477
Extractibility by petrol, %	3.15	4.27	1.48	1.47	1.44	1.44	1.45	1.44
Extractibility by oil, %	12.8	13.1	10.5	10.6	10.7	10.8	10.8	10.9
Melt flow index, g/10 min.	39.8	39.6	40.4	40.2	40.0	40.3	40.1	40.0
Heat stability at 175°C	169	167	175	173	170	173	172	172
Longterm film storage at 25°C during 4 month	Films are good							

at break was determined according to GOST (Standard-Setting Authority) 11262-80 at temperature (23±2)°C on preconditioned (GOST 12423-66) samples; a melt flow rate (MFR) according to GOST 11645-73 on extrusion type plastometer brand IIRT-5; while measurement of the brittleness temperature of PVC compounds were performed by express method in accordance with GOST 16782-92 in three samples which size is 130x10 mm and thickness is 1±0,1 mm; the thermal resistance of PVC compositions were evaluated according to GOST 14041-91 in terms of «time of heat stability». Time of heat stability was determined by time of an inductive discoloration of the «congo red» indicator at a temperature of 180°C and selections of HCl during polyvinylchloride destruction. Assessment of the technological properties of the developed PVC compounds was carried out with Brabender plastograph, rollers, twin-screw extruder MD 30-19, plastometer MMFT.

All tested samples of synthesized esters provided PVC films with relevant technical properties. Important technological parameters, such as time of heat stability, melt flow rate, in all cases of the use of test plasticizer samples were much higher, that indicates facilitated processing of the PVC compositions. It should be also noted that the PVC films have improved oil and petrol resistance. In addition, preliminary tests showed that obtained esters belong to the 3 hazard class. As it is known, DOP is a toxic plasticizer, which belongs to the 2 hazard class.

Thus, PVC compounds that contain the new plasticizers by all characteristics meet the existing standards and are recommended for further testing.

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НОВЫЕ ПЛАСТИФИКАТОРЫ ПОЛИВИНИЛХЛОРИДА

Аннотация к статье (авторское резюме, реферат):

Одним из основных крупнотоннажных полимеров современной химической промышленности является поливинилхлорид (ПВХ). Поливинилхлорид характеризуется многими полезными техническими свойствами – химической стойкостью в различных средах, хорошими электрическими свойствами и т. д. Это объясняет чрезвычайно разнообразное применение материалов на основе ПВХ в различных отраслях техники. Особенно широко применяется ПВХ в кабельной, строительной, легкой промышленности, в машиностроении, автомобилестроении и т. д. Одной из причин чрезвычайно быстрого роста производства ПВХ является то обстоятельство, что пока нет другого полимера, который можно было бы подвергать такому разнообразному модифицированию, как это делают с ПВХ.

Однако этот полимер при обычной температуре хрупок и неэластичен, что ограничивает область его применения. Быстрый рост производства поливинилхлорида объясняется его способностью к модификации свойств за счет введения при переработке специальных добавок. Введение в ПВХ пластифи-

каторов – в основном сложных эфиров органических и неорганических кислот – позволяет значительно изменить свойства полимера. Пластификаторы облегчают процесс получения полимерной композиции, увеличивают гибкость и упругость конечного полимерного продукта за счет внутренней модификации полимерной молекулы.

В работе приводятся результаты исследования методов получения, физико-химические и физико-механические свойства новых химикатов-добавок ПВХ (пластификаторов на основе оксиалкилированных спиртов). Приведены оптимальные условия синтеза соединений и результаты испытаний их в качестве добавок в рецептурах ПВХ-пленок. Отмечено, что композиции, полученные с введением в ПВХ-рецептуру предложенных пластификаторов, по всем показателям удовлетворяют требованиям действующих стандартов, а по показателям масло- и бензостойкости даже превосходят стандартные образцы.

Ключевые слова: оксиалкилированные бутанолы и фенолы, фталаты оксиалкилированных спиртов, пластификаторы ПВХ, ПВХ-пленка, маслостойкость, бензостойкость, относительное удлинение при разрыве, показатель текучести расплава, прочность при разрыве, температура хрупкости, термостабильность.

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