

UDC 678

**Author: MASKOVA Albina Rafitovna**, PhD in Engineering, Associate Professor of Department «Applied and Natural Sciences», Ufa State Petroleum Technological University; Mendeleev St., 195, Ufa, Bashkortostan Republic, Russia, 450080, asunasf@mail.ru;

**Author: AMINOVA Guliya Karamovna**, Doctor of Engineering, Professor of Department «Applied and Natural Sciences», Ufa State Petroleum Technological University; Mendeleev St., 195, Ufa, Bashkortostan Republic, Russia, 450080, aminovagk@inbox.ru;

**Author: FAIZULLINA Svetlana Radikovna**, Assistant of Department «Applied and Natural Sciences», Ufa State Petroleum Technological University; Mendeleev St., 195, Ufa, Bashkortostan Republic, Russia, 450080, sveta-mol86@mail.ru;

**Author: FAIZULLINA Galiya Fatykhovna**, Engineer of Department «Applied and Natural Sciences», Ufa State Petroleum Technological University; Mendeleev St., 195, Ufa, Bashkortostan Republic, Russia, 450080, galiya.aminova@gmail.com;

**Author: MAZITOVA Aliya Karamovna**, Doctor of Chemistry, Professor, Head of Department «Applied and Natural Sciences», Ufa State Petroleum Technological University; Mendeleev St., 195, Ufa, Bashkortostan Republic, Russia, 450080, elenaasf@yandex.ru

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## PRODUCTION OF PVC-FILMS WITH SPECIFIC PROPERTIES

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### EXTENDED ABSTRACT:

When developing polymeric materials with specified properties special compounds such as fungicides are used alongside traditional chemical additives (plasticizers). Usage of fungicides are determined by negative influence of mold fungi, bacteria and other microorganisms on polyvinyl chloride (PVC) material.

The present work focuses on the possibility to implement octylphenoxypropyl phthalates (OPOPP), proposed as plasticizers, and derivatives of 3-mercapto-1,2,4-triazinon-5, proposed as fungicides, in PVC films. The first additive was produced by esterification of phthalic anhydride by oxypropylated phenols and 2-ethylhexanol, the second additive – by cyclocondensation of alpha-keto acids with thiosemicarbazide.

Having analyzed the results of experiments the authors found out that compositions obtained with the use of new additives – octylphenoxypropyl phthalates and derivatives of 3-mercapto-1,2,4-triazinone-5, acquire high performance



properties that leads to increased service life. At the same time such important characteristics as strain at elongation, ultimate tensile stress, thermostability and funginertness are enhanced.

**Key words:** funginertness, dioctyl phthalate, tension at elongation, octylphenoxypropyl phthalate, ultimate tensile stress, PVC film, plasticizers of polyvinyl chloride, thermostability, fungicide.

DOI: [dx.doi.org/10.15828/2075-8545-2018-10-4-102-115](https://dx.doi.org/10.15828/2075-8545-2018-10-4-102-115)

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**P**olymeric materials are used in different fields of industry. One can notice the increasing number of the works devoted to polymers with specified characteristics, as well as advanced requirements to be met by these materials.

At the present time polyvinyl chloride (PVC) is the most widely used polymer. Pure polyvinyl chloride is difficult processed, for this reason PVC is mixed with different additives. The main role of chemical additives is to facilitate polymeric mixtures processing and giving the necessary properties to finished products. A wide range of physical and mechanical properties of plastics are obtained by using additives, that makes it possible to produce a wide variety of products – from very soft, gel-like to elastic, rigid materials. Thus chemical additives expand the field of PVC applica-



tion and allow it to be considered as a multicomponent system and the most easily compounded thermoplastic polymer [7–12].

More than 12 million tons of polymeric additives are produced every year in the world and plasticizers are the major part of them. Partial use of PVC in plasticized form are determined by characteristics of its chemicals structure – low elasticity, a significant temperature range of plastic deformation, and insufficient frost resistance [2, 5–7, 11, 12]. For this reason, intensification of manufacturing process and expansion of PVC application fields and providing wide variety of its properties, are connected to a large extent with the successful solution of the problem of creating effective plasticizers.

As known, phthalates are widely used as plasticizers in polymer industry. Dioctyl phthalate (DOP) is the main representative of phthalate plasticizers and it still remains the most widespread ester plasticizers of PVC. DOP possesses the optimal combination of properties, it is comparatively cheap, but DOP doesn't provide necessary service properties especially funginertness [11].

In this connection it is advantageous to produce the following additives: octylphenoxypropyl phthalates (OPOPP), which are proposed as plasticizers and derivatives of 3-mercapto-1,2,4-triazinone-5, which are proposed as fungicides. It is of great interest to study their properties and the possibility to develop PVC-materials that could meet the advanced operation requirements with the use of them.

We have produced new ester compounds – octylphenoxypropyl phthalates (OPOPP) with hydroxypropylation degree of phenol 1.0–2.1, that present slightly hygroscopic, oily liquids of yellowish color, highly soluble in organic solvents. OPOPP were obtained by the methods previously developed by us, by the esterification of phthalic anhydride by oxypropylated phenols and 2-ethylhexanol [13–27]. The physicochemical properties of proposed plasticizers are shown in table 1.

To determine the efficiency of suggested plasticizers the changing of Shor A hardness from the concentration of plasticizer were investigated (plasticizer: PVC – 1:100) [2-6]. On the base of calculation of quantitative substitution factor (SF) it was determined that 1,0-OPOPP is very similar to DOP by plasticization efficiency (table 1).

As known, plasticized PVC materials can be exposed to various microorganisms including fungus under unfavorable conditions which adversely



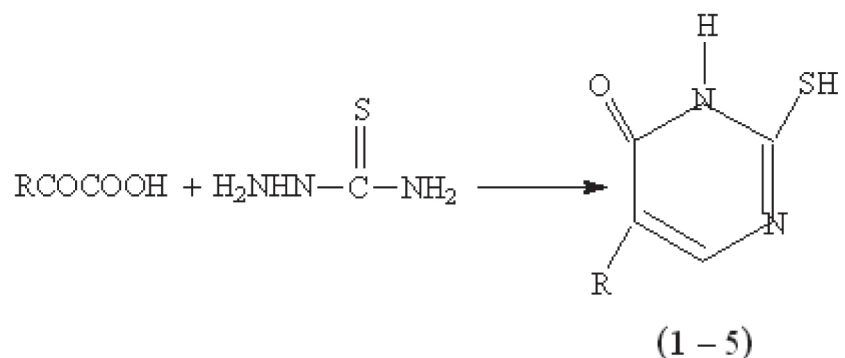
affected on the visual appearance of plastic compounds and reduce their service life. One of the way to protect PVC materials from negative influence of mold fungi is to introduce fungicides into compositions [11, 28].

Table 1

### Physicochemical properties of octylphenoxypropyl phthalates

Characteristics	1,0-OPOPP	1,5-OPOPP	2,1-OPOPP
Yield, %	89	87	84
Ester number., mg KOH/g	269	251	232
Molecular mass, found	416	446	481
Flash point, °C	199	198	198
Substitution factor	1,019	1,043	1,052

To realize this we obtained derivatives of 3-mercapto-1,2,4-triazinones-5 (scheme 1):



where R = CH<sub>3</sub> (1), i-C<sub>3</sub>H<sub>7</sub> (2), i-C<sub>4</sub>H<sub>9</sub> (3), t-C<sub>4</sub>H<sub>9</sub> (4), C<sub>6</sub>H<sub>5</sub> (5).

#### Scheme 1

Compounds of the heterocyclic series were obtained by cyclocondensation of alpha-keto acids with thiosemicarbazide.

At the beginning, the reagents were loaded in the following order: water 100 ml (solvent), sodium hydrogen carbonate 0,01 mole (cyclizing



agent), alpha-keto acid 0,1 mole, thiosemicarbazide 0,1 mole. The reaction was performed at 100°C for 3 hours then cooled to 10°C and neutralized with concentrated hydrochloric acid to pH = 6,0. The obtained precipitate was filtrated, washed with water and dried on the air.

For obtaining 3-mercapto-1,2,4-triazinon-5 with the maximum yield, the filtrate was treated with a concentrated solution of sodium hydroxide and kept under synthesis conditions for 1 hour. The resulting precipitate was filtered off, washed with water and air-dried.

3-Mercapto-1,2,4-triazinones-5 are solids, from white to orange colour, soluble in many organic solvents.

Physicochemical properties and spectral characteristics of the synthesized compounds are presented in table 2.

Table 2

**Physicochemical properties and spectral characteristics  
of 3-mercapto-1,2,4-triazinones-5**

Characteristics	3-Mercapto-1,2,4-triazinones-5				
	1	2	3	4	5
Yield, %	80	80	74	79	80
Melting point, °C	217–219	132–134	160–162	280–282	255–257
IR-spectrum, $\text{cm}^{-1}$	1670,1270, 1520	1675,1270, 1540	1690,1260, 1390	1675,1260, 1520	1670,1260, 1540
UV-spectrum, nm	190,275,310	195,280,319	190,270,315	192,270,320	220,290,330

Preliminary investigations have shown that derivatives of 3-mercapto-1,2,4-triazinon-5 possess high fungicidal activity [29, 30].

Next the produced compounds were tested in the PVC-films. Physical and mechanical properties of obtained PVC-films were analyzed according to standards: thermostability – according to GOST 14041-91, melt flow index – according to GOST 11645-73, funginertness – according to GOST 9.049-91, method 1.



Table 3

**The results of testing of plasticized PVC-films containing  
3-mercapto-1,2,4-triazinones-5 as additive**

Characteristics	Control*	1,0-OPOPP + 3-mercapto-1,2,4-triazinones-5				
		1	2	3	4	5
Strain at elongation 100%, MPa	11,1	11,9	11,8	12,0	12,2	12,4
Ultimate tensile stress, MPa	21,6	22,8	22,7	22,9	23,2	23,4
Breaking elongation, %	340	305	305	309	308	310
Extractability by waters, %	0,012	0,212	0,243	0,146	0,087	0,014
Water absorption, %	0,195	0,312	0,315	0,294	0,207	0,201
Melt flow rate, g/10min	44,5	40,2	40,4	39,3	38,9	38,7
Thermostability at 175°C, min	165	170	169	172	174	175
Funginertness, grade	Not re- sistant	Resis- tant	Resis- tant	Resis- tant	Resis- tant	Resis- tant

\* PVC-film, plasticized with DOP, without 3-mercapto-1,2,4-triazinones-5

As it can be seen from table 3, PVC-films produced by introducing developed octylphenoxypropyl phthalates and derivatives of 3-mercapto-1,2,4-triazinone-5, acquire high performance properties that leads to increased service life of the films. In particular, one should note the improvement of the following indices: strain at elongation, ultimate tensile stress and thermostability. Evaluating funginertness of materials by the degree of fungi it has been found out that composition prepared on the base of PVC with octylphenoxypropyl phthalates and derivatives of 3-mercapto-1,2,4-triazinone-5 acquire high fungi resistance, while compositions prepared with the use of only plasticizer DOP were unstable with respect to microorganisms.

All this indicates the perspective of using octylphenoxypropyl phthalates as plasticizers and derivatives of 3-mercapto-1,2,4-triazinone-5 as fungicides.





The performed research has been funded through the government grant «Synthesis and investigation of the modern polyvinyl chloride plasticizers» according to the Decree of the Government of the Republic of Bashkortostan of 07.02.2018 № 56 «On providing grants of the Republic of Bashkortostan to young scientists and groups of young researchers in 2018».

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*Maskova A.R., Aminova G.K., Faizullina S.R., Faizullina G. F., Mazitova A.K.* Production of PVC-films with specific properties. *Nanotehnologii v stroitel'stve = Nanotechnologies in Construction*. 2018, Vol. 10, no. 4, pp. 102–115. DOI: [dx.doi.org/10.15828/2075-8545-2018-10-4-102-115](https://doi.org/10.15828/2075-8545-2018-10-4-102-115).



УДК 678

**Автор: МАСКОВА Альбина Рафитовна**, к.т.н., доц. каф. «Прикладные и естественнонаучные дисциплины», ФГБОУ ВО «Уфимский государственный нефтяной технический университет»; ул. Менделеева, 195, г. Уфа, Республика Башкортостан, Россия, 450080, asunasf@mail.ru;

**Автор: АМИНОВА Гулия Карамовна**, д.т.н., проф. каф. «Прикладные и естественнонаучные дисциплины», ФГБОУ ВО «Уфимский государственный нефтяной технический университет»; ул. Менделеева, 195, г. Уфа, Республика Башкортостан, Россия, 450080, aminovagk@inbox.ru;

**Автор: ФАЙЗУЛЛИНА Светлана Радиковна**, ассистент каф. «Прикладные и естественнонаучные дисциплины», ФГБОУ ВО «Уфимский государственный нефтяной технический университет»; ул. Менделеева, 195, г. Уфа, Республика Башкортостан, Россия, 450080, sveta-mol86@mail.ru;

**Автор: ФАЙЗУЛЛИНА Галия Фатыховна**, инженер каф. «Прикладные и естественнонаучные дисциплины», ФГБОУ ВО «Уфимский государственный нефтяной технический университет»; ул. Менделеева, 195, г. Уфа, Республика Башкортостан, Россия, 450080, galiya.aminova@gmail.com;

**Автор: МАЗИТОВА Алия Карамовна**, д.х.н., проф., зав. каф. «Прикладные и естественнонаучные дисциплины», ФГБОУ ВО «Уфимский государственный нефтяной технический университет»; ул. Менделеева, 195, г. Уфа, Республика Башкортостан, Россия, 450080, elenaasf@yandex.ru

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## ПОЛУЧЕНИЕ ПВХ-ПЛЕНОК, ОБЛАДАЮЩИХ СПЕЦИФИЧЕСКИМИ СВОЙСТВАМИ

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### АННОТАЦИЯ К СТАТЬЕ (АВТОРСКОЕ РЕЗЮМЕ, РЕФЕРАТ):

При разработке полимерных материалов, обладающих специфическими свойствами, наряду с применением традиционных химических добавок (пластификаторов) используются также и такие специальные вещества, как фунгициды. Применение последних обусловлено негативным воздействием на поливинилхлоридные (ПВХ) материалы плесневых грибов, бактерий и других микроорганизмов.

В настоящей работе исследована возможность практического применения октилфеноксипропилфталатов (ОФОПФ), предложенных в качестве пластификаторов, и производных 3-меркапто-1,2,4-триазинов-5, предложенных в качестве фунгицидов, в ПВХ-пленках. Первая добавка получе-



на этерификацией фталевого ангидрида оксипропилированными фенолами и 2-этилгексанолам, вторая – циклоконденсацией альфа-кетокислот с тиосемикарбазидом.

На основании проведенных экспериментов было установлено, что композиции, полученные с использованием новых добавок, а именно октилфеноксипропилфталатов и производных 3-меркапто-1,2,4-триазинов-5, приобретают высокие эксплуатационные свойства, что приводит к увеличению срока их службы. При этом улучшаются основные показатели: напряжение при удлинении, разрушающее напряжение, термостабильность и грибостойкость.

**Ключевые слова:** грибостойкость, диоктилфталат, напряжение при удлинении, октилфеноксипропилфталат, разрушающее напряжение, ПВХ-пленка, пластификаторы поливинилхлорида, термостабильность, фунгициды.

DOI: [dx.doi.org/10.15828/2075-8545-2018-10-4-102-115](https://dx.doi.org/10.15828/2075-8545-2018-10-4-102-115)

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Работа выполнена при поддержке научного гранта «Синтез и исследование современных пластификаторов поливинилхлорида», в соответствии с Постановлением Правительства РБ от 07.02.2018 г. № 56 «О выделении в 2018 году грантов Республики Башкортостан молодым ученым и молодежным научным коллективам».



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*Маскова А.Р., Аминова Г.К., Файзуллина С.Р., Файзуллина Г.Ф., Мазитова А.К.* Получение ПВХ-пленок, обладающих специфическими свойствами // Нанотехнологии в строительстве. – 2018. – Том 10, № 4. – С. 102–115. – DOI: [dx.doi.org/10.15828/2075-8545-2018-10-4-102-115](http://dx.doi.org/10.15828/2075-8545-2018-10-4-102-115).

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*Maskova A.R., Aminova G.K., Faizullina S.R., Faizullina G. F., Mazitova A.K.* Production of PVC-films with specific properties. Nanotehnologii v stroitel'stve = Nanotechnologies in Construction. 2018, Vol. 10, no. 4, pp. 102–115. DOI: [dx.doi.org/10.15828/2075-8545-2018-10-4-102-115](http://dx.doi.org/10.15828/2075-8545-2018-10-4-102-115).

