

# The Use of Products of Microwave Aminolytic Destruction of Polyethylene Terephthalate in Vibration-absorbing Polymer Composite Materials

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**Abstract:** *The aim of this work is to study the possibility of using the products of aminolytic destruction of polyethylene terephthalate (PET) and their derivatives in polymer composite materials with vibration-absorbing properties, which provide effective damping of vibrations of technogenic and natural origin in a wide temperature and frequency range. The paper considers a modern method of recycling PET waste with a mixture of amino alcohols - monoethanolamine and triethanolamine, taken in two different ratios. As a result of the destruction reaction, terephthalic acid diamide (N, N'-bis (2-hydroxyethyl) terephthalamide) is formed. To accelerate the destruction process, microwave radiation of various powers of 200, 540 and 700 watts was used. The optimal conditions for aminolytic decomposition of PET were determined: the time and power of microwave radiation with PET conversion up to 95% and the yield of the target product (terephthalic acid diamide) 80-85%. The destruction process was carried out according to a closed cycle of using reagents, without the use of catalysts and at atmospheric pressure, which to a certain extent reduces the energy consumption and increases the environmental friendliness of this method of PET decomposition. The aminolytic degradation product of PET (terephthalic acid diamide) was used as a monomer in the polycondensation reaction to obtain a new oligomer (terephthalic acid oligoesteramide). The degree of polymerisation (n) is in this case 7 to 11 (number of chain links). The obtained oligomer and the PET degradation product were investigated as new components in elastomeric compositions based on chloroprene rubber and in compositions based on thermoplastic elastomers. It has been demonstrated that the introduction of an oligomer based on a PET degradation product reduces the viscosity of elastomeric compositions by 25-35%. The study was supported by a grant from the Russian Science Foundation No. 21-79-00301, <https://rscf.ru/project/21-79-00301/>.*

**Keywords:** *aminolysis, PET, microwave radiation, chloroprene rubber, vibration absorption*

## 1. Introduction

The processing of household or industrial solid waste of polyethylene terephthalate (PET) attracts more and more attention every year. This is due not only to the desire to reduce the environmental load on the environment, but also to the fact that PET processing products can be used in various industries [1], including as fillers or additives to vibration-absorbing materials operating in a wide temperature range.

Currently, there are several main methods for recycling PET waste: landfill, mechanical recycling (shredding and reuse), chemical recycling and incineration [2-5].

Chemical processing is one of the processing methods by chemical modification or deconstruction of polymer macromolecules. There are several solvolytic methods that are effective for PET deconstruction. These methods are: alcoholysis (reaction with alcohol) [6-11]; aminolysis (reaction with amines) [12-15]; ammonolysis (reaction with ammonia) and hydrolysis (reaction with water) [16-19]. The obtained compounds can be used as additives in asphalts and bitumen [20 - 22]; as a modifying or

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crosslinking additive in epoxy resins [23-25], to obtain protective coatings [26-28]; as one of the components of polyurethanes [29-32] and so on.

Solvotic methods of PET destruction can proceed under various conditions, with and without catalysts [33-37]. Destruction methods using microwave radiation instead of convective heating are of particular interest. Microwave radiation allows destruction processes to be carried out several times faster than with convective heating. This is primarily due to the fact that all reacting components in PET degradation reactions are polar compounds [38-42].

The obtained materials can be used as fillers for vibration-absorbing materials operating in a wide temperature range and used in transport [43-45] and in construction [46-48].

The aim of this work is to study the process of aminolytic destruction of PET waste under microwave radiation of various power. The effect of aminolytic degradation products of PET and their derivatives on sulfur vulcanization and elastic-strength properties of vibration-absorbing composite materials based on chloroprene rubber has been studied.

## 2. Materials and methods

For PET destruction, a mixture of amino alcohols was used - monoethanolamine (MEA) technical (TU 2423-159-00203335-2004) and triethanolamine (TEA) (TU 2423-168-00203335-2007) produced by Kazanorgsintez PJSC. PET waste is shredded plastic bottles without labels, with a particle size of 5x5 mm.

Study of the effect of the degradation product of PET and its derivatives of wires in formulations based on chloroprene rubber of three types of crystallization (controlled by mercaptans) CR121, CR232 and CR244, made in China, Shanxi Synthetic Rubber Group Co. Ltd. The formulations of model rubber compounds are presented in Table 1.

**Table 1.** Model rubber compounding

Ingredients	Mass parts per 100 mass parts of rubber, mass.p.		
	1 (CR232)	2 (CR244)	3 (CR121)
Rubber	100	100	100
Magnesium oxide	4	4	4
Zinc oxide	5	5	5
Sulfur	1	1	1
Tetramethylthiuram disulfide	1	1	1
Diphenylguanidine	1	1	1
Carbon black (P 803)	40	40	40
Total	160	160	160

Three mixtures with different brands of rubbers differing in the rate of crystallization were taken as basic.

Rubber mixtures were made on a laboratory micro-mixer with a loading chamber volume of 0.1 l (manufactured by Polimer mash Group LLC).

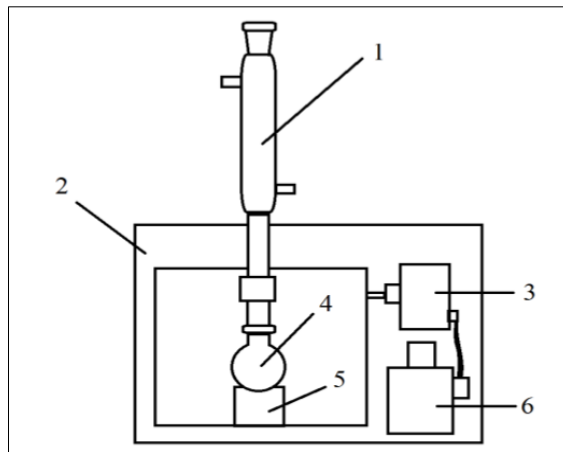
The vulcanization characteristics were measured using the Moving Die Rheometer equipment manufactured by Prescott Instruments Ltd. Mooney viscosity was determined on a Mooneyline MV Variable Speed rotational viscometer, Prescott Instruments Ltd. The determination of the elastic-strength properties was carried out on an AG-X 5 kN tensile testing machine, manufactured by Shimadzu.

The studies of the manufactured rubbers were carried out according to standard methods in accordance with GOST: removal of the vulcanization characteristics of rubber compounds (GOST 12535-84), determination of the viscosity of the crude rubber mixture according to Mooney on a rotary viscometer (GOST 10722-76), elastic-strength properties (GOST 270-75), rubber hardness according to Shore A (GOST 263-75), tests for accelerated aging and heat resistance (GOST ISO 188-2013).

### 3. Results and discussions

#### 3.1. Kinetics of aminolytic destruction of polyethylene terephthalate by microwave radiation

The laboratory setup for the aminolytic destruction of PET was based on a household microwave oven with the ability to control the microwave radiation power (Figure 1). The main major design change is the ability to connect a reflux condenser to a reaction flask inside the microwave.



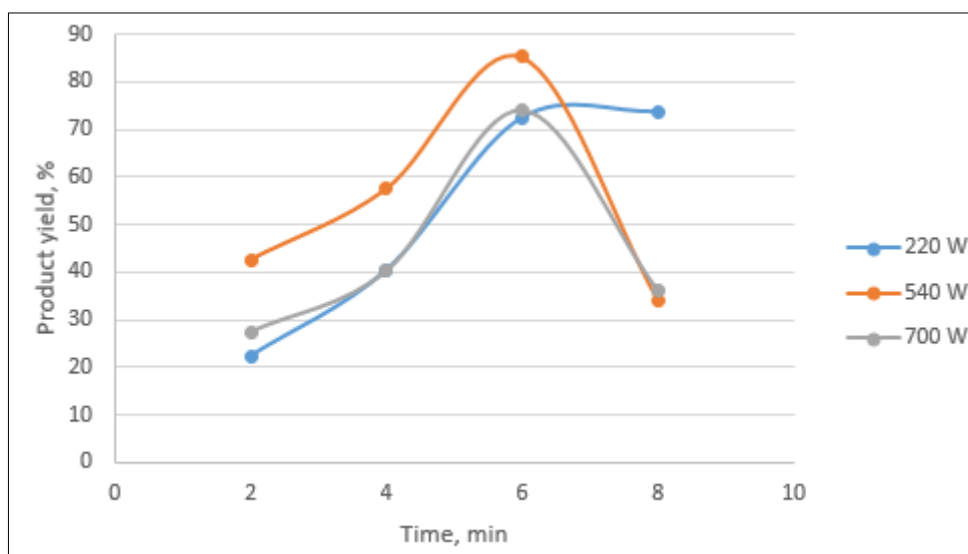
**Figure 1.** Schematic of a laboratory setup for PET destruction under microwave radiation (1 - reverse cooler; 2 - installation body; 3 - magnetron; 4 - reaction flask; 5 - stand; 6 - transformer)

Aminolytic destruction of PET particles (particle size 5x5 millimeters) was carried out in a mixture of amino alcohols in a ratio of 1: 4: 5 and 1: 3: 4, respectively, for PET: MEA: TEA. The choice of these ratios of amino alcohols is due to the need for a sufficient amount of amino alcohols to degrade PET particles. PET weight in these cases is 10 g. The kinetics of PET destruction at different microwave power (220, 540 and 700 watts) and two component ratios are shown in Figures 2 and 3.

The yield of BHETA was calculated using (1):

$$\text{Yield}_{\text{BHETA}}(\%) = M_{\text{BHETA}}/M_{\text{BHETA0}}, \quad (1)$$

where  $M_{\text{BHETA}}$  and  $M_{\text{BHETA0}}$  are the experimental weight of BHETA and the theoretical weight of BHETA that would be produced upon complete decomposition of PET, respectively



**Figure 2.** Kinetics of PET destruction at a component ratio of 1:4:5 (PET:MEA:TEA)

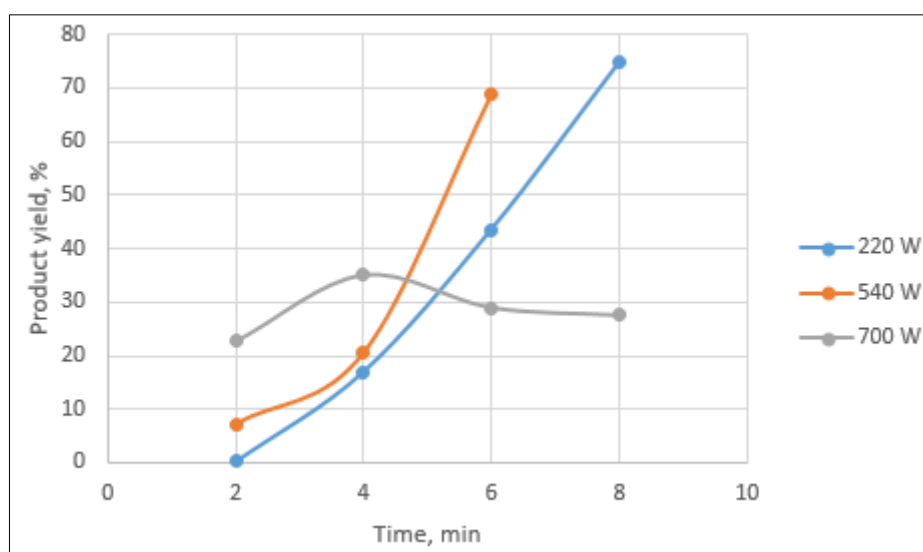
From the data presented in Figure 2, it can be seen that the maximum product yield, 85%, is achieved with a reaction time of 6 min and a microwave power of 540 W. With a longer time of exposure to microwave radiation under these conditions, the product yield decreases due to significant evaporation of the reaction mass (a mixture of amino alcohols) and its presence at the junction of the reflux condenser and the reaction flask in the form of vapors.

When using a power of 220 W, the highest product yield (84-86%) is achieved at 8 min of reaction, which is associated with a slow heating of the reaction mixture and a decrease in the rate of evaporation of the mixture of amino alcohols.

The use of a radiation power of 700 W is impractical due to too rapid heating, boiling and evaporation of the reaction mass. The contact time of the mixture of amino alcohols with PET particles is short, which leads to a lower product yield (up to 74%).

When the ratio of the components is 1: 3: 4, there is a general decrease in the yield of the target product at all utilized capacities (Figure 3). In this case, the maximum product yield reaches only 74%. The decrease in the product yield is due to the absence of MEA in the mixture of amino alcohols, which, under the action of microwave radiation, especially at a power of 700 W, evaporates very quickly and does not have time to completely condense when it enters the reverse condenser. As a consequence of this, the concentration of MEA in the reaction mass decreases very quickly.

After the destruction process, the resulting product was purified by double recrystallization from hot water. The products obtained were identified by NMR analysis ( $^1\text{H}$  and  $^{13}\text{C}$ ), gas chromatography-mass spectrometry (at a temperature of  $500^\circ\text{C}$ ) and FT-IR spectroscopy.



**Figure 3.** Kinetics of PET destruction at a component ratio of 1:3:4 (PET:MEA:TEA)

### 3.2. Obtaining an oligomer based on a PET degradation product

The PET degradation product (terephthalic acid diamide) was used as a monomer in the polycondensation reaction to obtain an oligomer. The reaction was carried out in the temperature range from 200 to  $215^\circ\text{C}$ , with evacuation to remove reaction by-products (ethanolamine). Phosphoric acid was used as a catalyst.

As a result of the reaction, a light yellow resinous substance was obtained, which crystallizes upon cooling, with a slight odor of amino alcohols. The color and odor of the resulting product is due to incomplete removal of polycondensation by-products of terephthalic acid diamide, namely monoethanolamine.

The resulting oligomer is soluble in highly polar solvents - dimethyl sulfoxide and dimethylformamide; has a good ability to stretch into fibers (up to several meters) in a warm and hot state. The

melting point of the resulting product is in the range from 75 to 80°C.

### 3.3. Chloroprene rubber compounds

Rubber compounds based on three types of chloroprene rubbers with different crystallization rates were made in accordance with the formulations shown in Table 2.

**Table 2.** Chloroprene rubber based rubber formulations

Ingredients	The rubber compound of the masses. part					
	4	5	6	7	8	9
Rubber	100					
Magnesium oxide	4					
Zinc oxide	5					
Sulfur	1					
Tetramethylthiuram disulfide	1					
Diphenylguanidine	1					
Carbon black (P 803)	39					
BHETA	1			-		
oATPA	-			1		
Total	152					
Note:						
*BHETA is a product of aminolytic degradation of PET						
** oATPA - oligomer based on PET degradation product						

Rubber compounds 4 and 7 are made on the basis of CR232 rubber; mixtures 5 and 8 - CR244; mixtures 6 and 9 - CR121.

### 3.4. Kinetics of vulcanization of rubber compounds

First of all, the influence of terephthalic acid diamide and an oligomer based on it on the kinetics of sulfur vulcanization of rubber compounds was considered. Table 3 presents data on the key vulcanization characteristics of sulfur vulcanization of the investigated rubbers T<sub>10</sub>, T<sub>50</sub> and T<sub>90</sub>.

**Table 3.** The duration of the periods of vulcanization of rubber compounds

Parameter	Compound								
	Modeling			4	5	6	7	8	9
	1	2	3						
T <sub>10%</sub> , s	87	57	74	91	60	84	93	84	69
T <sub>50%</sub> , s	220	226	148	265	229	149	278	242	158
T <sub>90%</sub> , s	1107	967	833	1082	953	797	1094	961	723

According to the data in Table 3, it can be seen that the accelerating effect on the sulfur vulcanization process was observed in almost all cases with the addition of terephthalic acid diamide and an oligomer based on it, which is especially clearly seen in the T<sub>90</sub> parameter. In mixtures with the addition of the PET degradation product, T<sub>90</sub> decreased by 1.5-4%, and in mixtures with the addition of an oligomer based on terephthalic acid diamide, T<sub>90</sub> decreased by 0.5-13%. The strongest reduction in T<sub>90</sub> was observed in mixture 9 - by 13.2% (reduction in vulcanization time by 2 min).

Such an accelerating effect on the sulfur vulcanization process may be due to the fact that the added ingredients (terephthalic acid diamide and oligomer based on it) are alkaline in nature.

In the case of parameters T<sub>10</sub> and T<sub>50</sub>, a slight (within 5%) slowdown of the process was observed, which leads to an increase in the induction period of vulcanization.

### 3.5. Elastic-strength properties of the obtained rubbers

Subsequently, the viscosity of raw rubber mixtures, as well as the elastic-strength characteristics of vulcanizates, were investigated. The vulcanization of rubbers took place at the optimum vulcanization at a temperature of 160°C for 15-17 min.

In addition, in addition to the main elastic-strength properties of rubbers, the influence of terephthalic acid diamide and an oligomer based on it on the aging process of rubbers was evaluated. Table 4 shows data on the viscosity of raw rubber compounds, as well as some indicators of elastic-strength properties of rubbers before and after thermal aging for 24 h.

**Table 4.** Elastic-strength characteristics of vulcanizates before and after thermal aging

Indicator / rubber compound	1	2	3	4	5	6	7	8	9
Viscosity, Mooney units	69	90	79	70	83	75	70	79	72
Hardness, Shore A	89	-	51	102	-	32	77	-	39
fp, MPa	20.1	24.7	18.8	20.6	23.5	18.1	21.7	25.9	18.9
$\epsilon_p$ , %	330	210	240	310	240	290	350	280	310
Stress at 100%, MPa	4.3	12.8	7.2	5.2	10.7	5.6	4.5	9.8	5.13
Stress at 200%, MPa	10.7	22.3	16.3	12.9	21.5	12.5	11.2	21.0	11.7
<b>After aging (24 h at 100°C)</b>									
fp, MPa	19.1	21.7	16.6	20.2	24.0	17.7	20.1	21.5	17.9
$\epsilon_p$ , %	290	220	260	360	300	260	310	270	270

From the data in Table 4 (study of elastic-strength properties) it can be seen that with the addition of terephthalic acid diamide, no significant changes in the relative strength at break were observed, this indicator remained at the level of the base mixtures of comparison. However, the addition of this component had an effect on the elongation at break: an increase in this indicator was observed for mixtures 5 and 6, especially in the case of the latter mixture, the increase was more than 50%.

In the case of using an oligomer based on terephthalic acid diamide, the relative strength of mixtures based on chloroprene rubbers CR232 and CR244 increased, and in the case of CR121 rubber it remained comparable to the base mixture. The relative elongation at break of the studied mixtures in all three cases increased by 20 to 70%, which indicates the plasticizing effect of the obtained oligomer.

In addition, the plasticizing effect of the obtained oligomer is confirmed by the data on the viscosity of rubber compounds. The viscosity of crude rubber compounds 7 and 9 is reduced by 13 and 24%, respectively.

Comparing the elastic-strength parameters of the obtained vulcanizates before and after aging, we can say that in some cases (mixtures of 4 and 5 with the addition of terephthalic acid diamide), an increase in the elongation at rupture of vulcanizates after aging was observed by 49 and 65%, respectively.

The conditional tensile strength after aging in vulcanizates with the addition of terephthalic acid diamide decreased to a lesser extent in comparison with the model mixtures: the decrease in the conventional strength at break in model mixtures is about 13%, in mixtures with the addition of terephthalic acid diamide 2%. This may be due to the fact that terephthalic acid diamide acts as an inhibitor of the thermooxidative process of chloroprene rubber due to the presence of an amide group.

## 4. Conclusions

The process of aminolytic destruction of polyethylene terephthalate waste was studied under microwave radiation of different power and with different use of components. The most optimal conditions for PET destruction under microwave radiation with a product yield of 82-85% and a destruction time of 4-6 min have been revealed.

The possibility of using the product of aminolytic destruction of PET and its oligomer in new ingredients as vibration-absorbing composite materials based on chloroprene rubber is considered. Some elastic-strength characteristics and the nature of their change with the addition of new ingredients. It was revealed that the new ingredients slightly accelerate the process of sulfur vulcanization of rubbers based on chloroprene rubbers; have a plasticizing effect on the studied vulcanizates, and can also act as inhibitors of thermo-oxidative processes.



A more detailed consideration of the influence of the new results obtained on the basis of polar rubbers and thermoplastics on the physicochemical and physicomechanical parameters is proposed.

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