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DETERMINATION OF THE INFLUENCE OF THE THERMOSTABILIZER CONTENT ON THE CYCLIC PROCESSING OF POLYVINYL CHLORIDE

The object of the study is the process of cyclic recycling of polyvinyl chloride (PVC). The main problem of multiple recycling of polyvinyl chloride is poor thermal stability. This problem is associated with insufficient stabilization of polyvinyl chloride at the stage of manufacturing the initial product. Usually, such products are difficult to recycle, since the content of additives in polyvinyl chloride is designed for one-time manufacturing of products and does not provide for repeated cyclic recycling. In addition, it may be necessary to modify the PVC composition to obtain the characteristics necessary for a new application. The thermal stability of polyvinyl chloride can be improved by various methods of physical modification. One of the key methods is the introduction of a stabilizer in a rational amount. To solve the problems set in the work, PVC composites with an adjustable stabilizer content from 2 to 5 mass parts were manufactured. The manufactured composites were recycled up to 5 times. At each cycle of PVC composites processing, thermal stability and melting point were determined using the RM-200C Hapro rheometer plastograph (Harbin University of Science and Technology Harbin Technology Co. Ltd, China). Mechanical properties were also determined on a tensile machine according to ISO 527-2:2012, Charpy impact strength according to ISO 179 and ASTM D256, and melt flow index according to ISO 1133:199.

The study found that increasing the stabilizer content by only 1–2 parts by mass from its base concentration of 3 parts by mass increases the life of the PVC composite by 1.5–2 times. A stabilizer content of 4–5 parts by mass allows maintaining sufficiently stable physical and mechanical characteristics of the PVC composite during 5 processing cycles.

As a result of the research, a PVC formulation suitable for cyclic processing was proposed, which is achieved by introducing 4–5 mass parts of the stabilizer at the stage of primary PVC production. This allows maintaining the mechanical characteristics of the material, valuable for the final application during multiple processing compared to typical PVC composites used on the market for the production of window profiles.

Keywords: *thermal degradation, rheometric analysis, plastogram, tensile strength, impact strength, melt flow index, extrusion.*

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1. Introduction

Polyvinyl chloride (PVC) is one of the oldest thermoplastic polymers. Since the beginning of the industrial synthesis of PVC in the early 1930s, its production has been constantly increasing [1]. It currently ranks third in the world in terms of production volume [2].

The widespread use of PVC is a consequence not only of its low production costs, but is primarily determined by its operational properties. PVC has good chemical resistance [3], high impact strength, abrasion resistance, good adhesive properties [4], high transparency, good electrical insulation properties, is non-flammable, and is also resistant to weather conditions [5]. The use of plasticizers allows for the production of hard and soft versions of PVC products, which differ significantly in glass transition temperature and flexibility at a certain operating temperature [6].

Approximately 70 % of PVC production is used in the construction industry, mainly as window and door profiles, water and sewer pipes, cable insulation, gutters, floor underlayment and roofing membranes [7].

However, despite the numerous advantages of PVC and a wide range of applications, this polymeric material has one important drawback – poor thermal stability. PVC begins to degrade at 140 °C, although its processing mainly occurs at 160–200 °C. To prevent the degradation process of PVC during processing, heat stabilizers are used. PVC heat stabilizers absorb and neutralize hydrogen chloride, and inhibit its autocatalytic action. The main classes of heat stabilizers currently used are metal salts, metal soaps and organotin compounds. Also, most stabilizers have a lubricating effect, reducing mechanical wear and extending the service life of equipment.

Despite the advantages and disadvantages of PVC products, their long-term service life, which can be decades, PVC waste is constantly increasing and requires constant recycling.

Methods of PVC recycling. There are several main areas of PVC recycling: incineration, thermal and mechanical processing of waste. Incineration of PVC waste is the process of thermal decomposition of polyvinyl chloride at high temperatures with energy recovery. However, this process is accompanied by a number of complex environmental problems. Thermal processing consists of decomposition into chlorine-containing components, which are subsequently used for re-synthesis of PVC or used in other industries. Mechanical processing consists of mechanical processing of PVC waste, namely cleaning and grinding. Then these wastes are usually added to the primary raw materials in an amount of no more than 30 %. The easiest way is to recycle the material directly at the production plant where the waste is generated. It is somewhat more difficult to obtain a consistent raw material composition when processing PVC products and materials. In this case, the need for raw material purification should be taken into account. In addition, it may be necessary to modify the PVC composition to obtain the specific processing and characteristics required for the new application.

Mechanical processing. PVC is considered a polymer material with very limited mechanical processing. This is due to the misconception of low thermal stability and hazardous decomposition products that cause increased corrosion of process equipment. The correct use of heat stabilizers allows for a PVC material with a very long thermal stability time, and therefore proper processing [4, 8].

When considering PVC recycling, it should be taken into account that in the processed PVC mixtures, in addition to the above-mentioned heat stabilizers, a number of other additives are used. These are external and internal lubricants, flow modifiers, mechanical property modifiers, plasticizers, and often a relatively high content of mineral fillers, such as chalk, talc and titanium whites. Their use allows for precise control of the technological and functional properties of the processed PVC mixture [6, 9].

In some cases, recycling of PVC material can save up to 90 % of energy compared to the energy consumption required for the use of virgin materials. Thus, carbon dioxide emissions are reduced [10].

Waste with a certain composition, which is mainly generated at the recycling plant, can be directly recycled by shredding. It has been proven that unplasticized PVC can be processed several times without obvious signs of degradation. Furthermore, the number of times the same material can be processed can be significantly increased by mixing recyclate with virgin material in amounts exceeding 30 % [11, 12].

Shredded PVC waste can be directly processed into other products. For technical reasons, it can be processed into pellets, although each subsequent processing step can reduce the thermal stability of PVC [11].

Incineration and thermal processing are alternatives to mechanical processing and post-consumer waste disposal. This method is more suitable for unsorted PVC waste streams for which material recycling is not possible or not economical. The main aim is to reintroduce the raw material into a closed loop and recover the energy contained in the material. The chemicals produced during the PVC decomposition have a variety of applications, especially in the chlorine industry [10].

Thermal recycling of PVC waste involves thermal treatment of the waste stream to recover hydrogen chloride, which is recycled for PVC production or other processes. PVC is a material for which thermal recycling has been found to be inefficient and therefore not suitable for the future. However, there is currently a great deal of intensive work being done on thermal recycling of this waste. Several thermal recycling processes are used, such as pyrolysis, gasification, incineration and their modifications. Many of the problems in thermal recycling are caused by process additives such as stabilizers and plasticizers commonly used in PVC recycling, which are currently on the list of banned substances [13].

Incorrect thermal recycling of Cl-containing waste, including PVC, can cause significant damage to installations due to the corrosive properties of the gaseous products formed. The formation of dioxins at inappropriate temperatures is also dangerous, which is why process control is so important.

The growing amount of PVC waste every year increasingly raises the issue of its recycling and implementation within the framework of a circular economy. Therefore, the current issue is the creation of new approaches to the process of recycling PVC waste.

The production of PVC products cannot do without the use of stabilizers. Stabilizers prevent the destruction of PVC, which occurs at temperatures above 130–140 °C, acting as a lubricant. However, excessive content of the stabilizer makes recycling impossible due to excessive lubrication. The stabilizer is an expensive additive, so its content is tried to be reduced to a minimum. Such a strategy leads to impossible or complicated re-processing, not to mention cyclic processing. An important aspect is to determine the necessary rational concentration of technological additives, primarily the stabilizer, which will ensure cyclic processing of PVC.

Therefore, *the aim of research* is to determine the rational content of calcium/zinc heat stabilizer at different stages of cyclic processing of PVC.

2. Materials and Methods

The work used polyvinyl chloride brand VYNOVA S6706 (Vynova Group, Belgium), calcium carbonate Omyacarb 2T-KA (KARABIGA, Turkey), calcium/zinc stabilizer BAEROPAN MC 91424 FP/1 (manufacturer Baerlocher Kimya San. Tic. Ltd. Şti. Akhisar Şubesi, Turkey), polyethylene wax Licowax PE 520 (Clariant, Germany). The research was conducted on the basis of the basic formulation of the PVC composition (Table 1). Such a PVC composition with the addition of processing modifiers, impact resistance and titanium dioxide are used in the manufacture of window profiles.

Table 1

Basic formulation of the PVC composition

Component	Brand	Mass parts
PVC	S6706	100
Calcium carbonate	Omyacarb 2T-KA	5
Calcium/zinc stabilizer	BP MC 91424 FP/1	3
Polyethylene wax	Licowax PE 520	0.5

In the basic formulation, the stabilizer concentration was adjusted by decreasing it to 2, and increasing it to 4 and 5 parts by mass, to determine the rational parameters of the additive under conditions of repeated processing.

2.1. Preparation of PVC composite samples and their cyclic processing

Preparation and cyclic processing of PVC composite samples took place in the following sequence. First, the initial components according to the selected formulation were mixed in a high-speed mixer Hurakan HKN-SG400 (Hurakan, China). PVC granulate was obtained from the powder mass on an extruder SJ25 (Nantong Bogda Machinery Technology Co., Ltd., China). A tape was formed from the granulate on the extruder. The tape was examined, and then crushed and reprocessed 5 times on the extruder again into a tape.

2.1.1. Production of PVC composite granulate

First, the PVC components of the composition are mixed in a high-speed Hurakan HKN-SG400 grinder (Hurakan, China). According to the recipe, PVC powder and stabilizer are loaded into the grinder and mixed for 3 min at a speed of 10–15 thousand rpm, after which all other components of the recipe are added and mixed for another 2 min. Then the powder mass was kept for 2 h before further processing. The resulting powder mass is extruded. The work used an extruder with a screw diameter of 25 mm and $L/d=16$ with a slotted loading zone and a die head with a diameter of 3 mm. The extruder temperature regime: loading zone – 165 °C, melting zone 185 °C and dosing zone 180 °C. The extruded strand is cooled in air, and then, using a pulling device, it enters the strand granulator. As a result of grinding, it is possible to obtain a granule approximately 2 by 2 mm.

2.1.2. Production of PVC composites in the form of a tape

The resulting PVC granulate is dried at a temperature of 75 °C for 5 hours. The dried granulate is processed into a tape on an extruder with a flat slot head. The temperature regimes of the extruder: loading zone – 165 °C, melting zone 185 °C and dosing zone 180 °C. After forming the strand on the extruder head, it is cooled in a water bath, and then wound onto a reel. As a result, let's obtain a tape with a thickness of 4 mm and a width of 18 mm.

2.1.3. Cyclic processing of PVC composite in the form of a tape

For further research, PVC tapes were crushed into granulate on a rotary knife crusher with a diameter of 4 mm of calibration mesh holes. Then the crushed PVC composite is dried at a temperature of 75 °C for 5 hours and processed again in an extruder into a tape. The tape was processed again 5 times.

2.2. Rheometric analysis

The obtained PVC composites in the form of granules were mixed in the chamber of the RM-200C rheometer (Harbin University of Science and Technology Harbin Electrical Engineering Co. Ltd, China), which simulates the conditions of PVC processing processes. In a closed heated chamber, the mixture was moved by two horizontal profiled roller-type rotors. During the test, the following parameters were recorded: the actual temperature of the mixed mixture and the torque of the rotors as a function of time in the form of a diagram called a plastogram. The plastogram allows to evaluate the general technological characteristics of the PVC compound and select rational processing parameters. A typical plastogram for a rigid PVC compound with the indicated critical points is shown in Fig. 1.

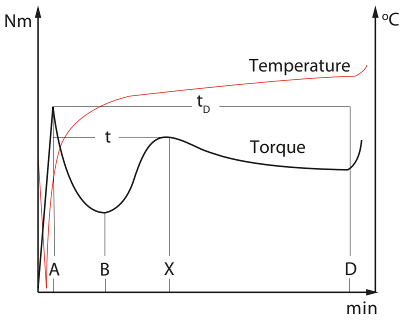


Fig. 1. Typical plastogram for rigid polyvinyl chloride compositions: A is the peak of loading, B is the minimum, X is the maximum, D is the decomposition, t is the synthesis time, t_D is the decomposition time

Point A is the peak of loading or synthesis. The point on the graph where the viscosity or resistance to deformation of the material reaches its maximum value at the initial loading (or at the initial stage of processing). It indicates a high resistance of the material to deformation at low temperatures. A high peak at the beginning of the plastogram means that the material is in a solid state, and for further processing it is necessary to reach a temperature at which the PVC begins to soften. This point is often an indicator for controlling the starting parameters of the extrusion. PVC welding is primarily affected by lubricants and process additives. External lubricant will increase the welding time and reduce the torque and temperature, vice versa for internal lubricants and process additives.

Point B is the minimum load on the plastogram corresponds to the point where the viscosity reaches its lowest value, i. e. when the polymer softens and becomes plastic. This is the best moment to shape the PVC material (extrusion, casting, stretching), as the material acquires the most suitable properties for processing. This occurs in the temperature range when the PVC molecules are mobile enough to flow, but have not yet reached overheating or degradation. Torque and temperature are in equilibrium. The equilibrium conditions can be affected by almost anything in the formulation.

Point X is the maximum on the plastogram can indicate the point where the material reaches its maximum fluidity before decomposition or degradation begins. This is the point where the material is most fluid and ready for further processing. The maximum usually indicates the optimal state for further processing (e. g. for extrusion or molding) and is critical for achieving high quality products. This maximum usually corresponds to the softening or melting temperature of the material.

Point D is the point at which the material begins to degrade. PVC begins to decompose or degrade due to overheating or prolonged exposure to high temperatures. If the temperature continues to rise after point X and reaches point D, the viscosity of the material will begin to increase again due to polymer degradation. This leads to a deterioration in the quality of the material and it can no longer be processed normally. In the extrusion or other processing process, reaching the degradation point leads to the formation of defects in the products and a decrease in mechanical properties.

t is the synthesis time (or processing time) indicates the duration of the PVC processing process under certain temperature conditions. The synthesis time allows to understand how long it takes for PVC to go through all the heating stages and reach the desired plasticity. Excessively long times can lead to material degradation, which will affect the final quality of the product.

t_D is the decomposition time determines how long the material can remain at high temperatures before it begins to degrade.

The stability of the compound during processing is determined by the effectiveness of additives (thermal stabilizers) and/or the temperature of the starting material.

2.3. Rheological characteristics

Melt flow index (MFI) is the rate of flow of polymer melt through a standardized capillary at a specified temperature. The MFI measurement process was carried out according to ASTM D1238 or ISO R1133. A constant pressure capillary viscometer of the IIRT-AM type (ASMA-Prybor LLC, Ukraine) was used to determine MFI in the work. The MFI of the polymer is determined by the following formula:

$$MFI = \frac{600\cdot\sigma}{t}\ \left(\mathrm{g/10\ min}\right), \tag{1}$$

where σ is the mass of the sample; t is the time interval between consecutive sections, s.

The MFI was determined on a constant pressure capillary viscometer at a temperature of (170±0.5) °C and a load mass of 5.0 kg.

2.4. Determination of mechanical properties under tension

The determination of mechanical properties under tension was carried out according to ISO 527-2:2012 on a ZD-10t/91M tensile machine (OOO PTP "ASMA-Prybor", Ukraine).

The value of the tensile strength σ_{pp} in MPa (N/m^2) is calculated by the formula:

$$\sigma_{pp} = \frac{F_{pp}}{A_0}, \tag{2}$$

where F_{pp} is the load at which the sample collapsed; A_0 is the initial cross-sectional area of the sample, mm^2.

The result is taken as the arithmetic mean of at least 5 determinations. The value of the relative elongation at break (ε_p, %) is calculated by the formula:

$$\varepsilon_p = \frac{\Delta l_p}{l_0}\cdot 100, \tag{3}$$

where Δl_p is the increase in the calculated length of the sample at the moment of rupture, mm; l_0 is the initial calculated length of the sample, mm.

2.5. Impact strength

The Charpy impact strength was determined according to ISO 179 and ASTM D256 standards. Using a pendulum impact tester MK-30 (ASMA-Prybor LLC, Ukraine).

3. Results and Discussion

PVC stabilization during processing plays an important role in the formation of the final product. Insufficient stabilization leads to thermal destruction of PVC and rapid wear of equipment. Excessive stabilization leads to excessive financial costs, and also makes it impossible to obtain finished products due to excessive lubrication of PVC powder during processing. However, most manufacturers of PVC products try to reduce the stabilizer content as much as possible. This strategy allows for one-time processing of PVC into products, but makes it impossible to recycle it. Taking this into account, the work conducted a study of the influence of the content of stabilizing additives at the stage of PVC production from primary materials – primary stabilization. With proper processing of PVC waste, photolytic and destructive damage to the material should not occur. The effect of the thermal stabilizer consists in the acceptance of the effect on hydroperoxides, deactivation of radicals, and adsorption of hydrogen chloride. After the first processing cycle, only a certain portion of the stabilizer is consumed, the remaining portion may be sufficient for further processing. The remaining amount of stabilizer may not be sufficient for further processing if its dosage was calculated for one processing cycle in a sparing mode. When processing rigid PVC waste, rational results are obtained by adding a finely ground mixture of stabilizers that simultaneously have a lubricating effect. In the basic PVC composite recipe, the stabilizer content was varied, reducing and increasing its content to 2 and 4.5 mass parts, respectively. Each of the resulting PVC composites was tested for 4 cycles on a rheometer. The results of the studies are shown in plastograms (Fig. 2–5).

According to the obtained plastograms in Fig. 2–5, it can be concluded that the increase in thermal stability of the basic PVC composite and its life time to 6–7 min occurs when the stabilizer content increases to 4–5 mass parts. At a lower stabilizer concentration, degradation occurs at 3–4 minutes of processing. It is worth noting that the stabilizer content at the level of 4–5 mass parts allows maintaining the physical and mechanical characteristics of the PVC composite at almost the same level during cyclic processing (Table 2).

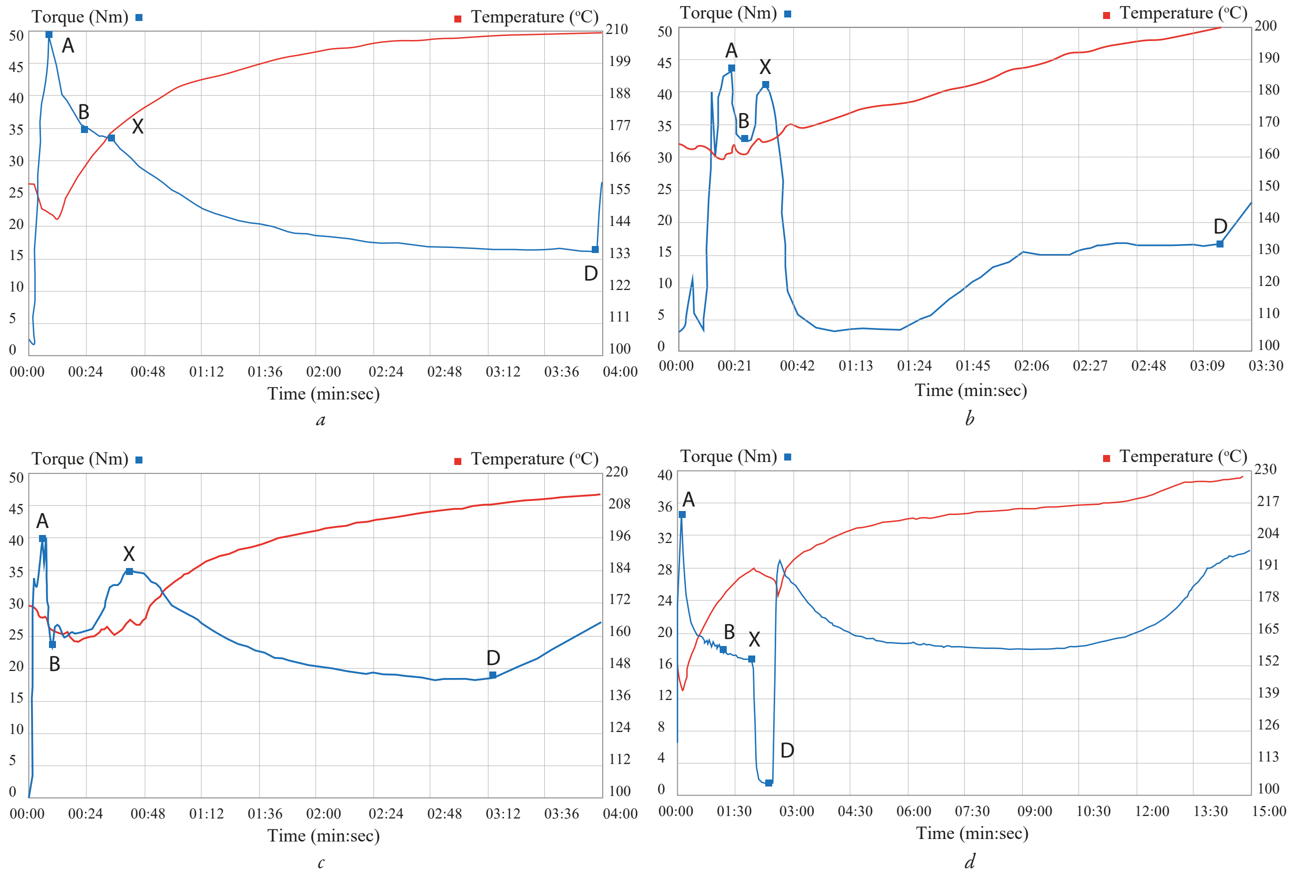
In the first recycling cycle, PVC has MFI index that corresponds to its typical characteristics, since the polymer molecules have a high molecular weight and retain good fluidity when heated. After 5 recycling cycles, the MFI index increases, probably due to a decrease in the molecular weight of the polymer.

The tensile strength of PVC decreases with repeated recycling due to a slight decrease in molecular weight with a slight degradation process.

The elongation during repeated recycling of PVC decreases, but not significantly due to a slight decrease in molecular weight, which indicates sufficient stabilization of the PVC composite and a low degree of degradation.

During repeated recycling of rigid PVC in a single-screw extruder at 185 °C, the impact strength increases until the third recycling cycle, and then remains constant.

Practical significance. The rational content of the heat stabilizer has been determined, which allows for repeated recycling of PVC composites, satisfying the conditions of the cyclical economy.



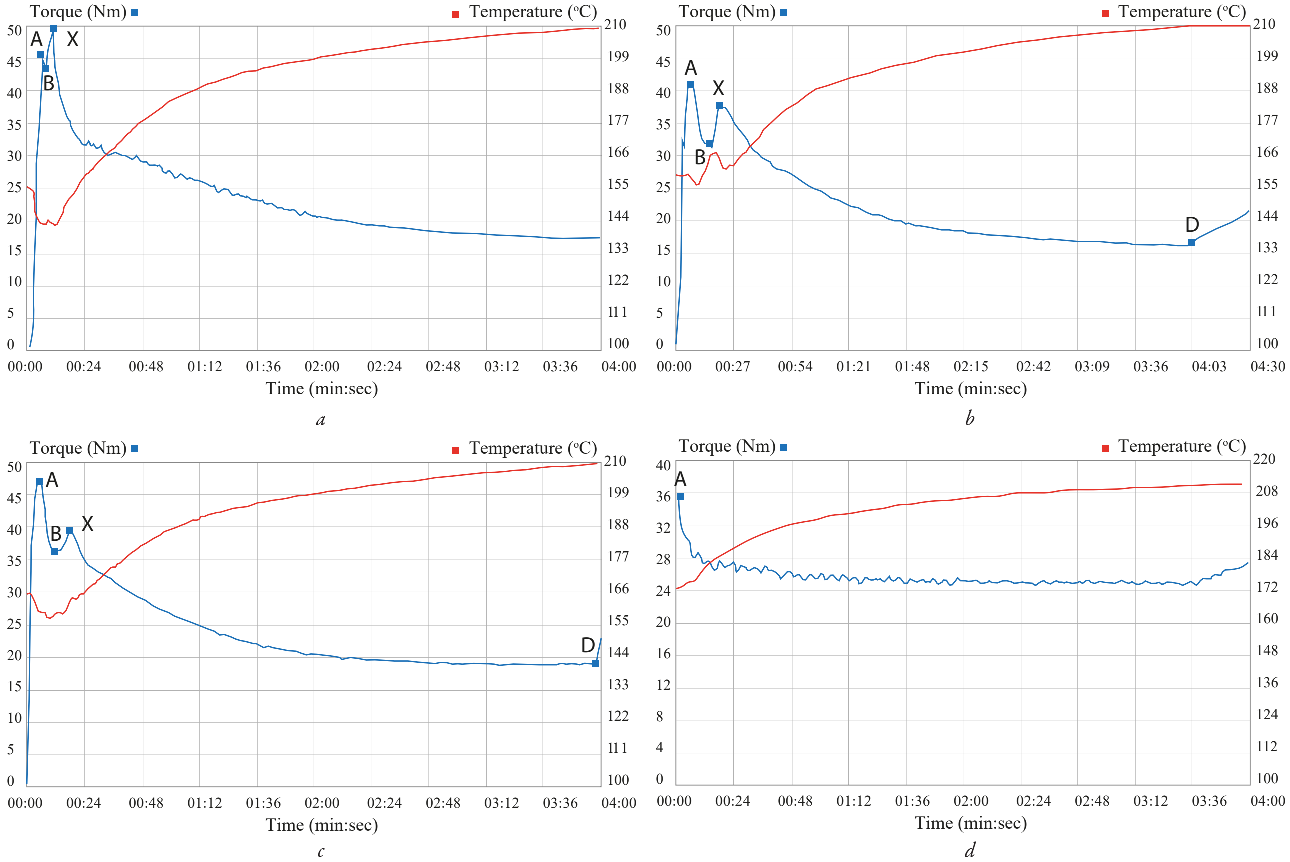
a

b

c

d

Fig. 2. Plastogram of the basic polyvinyl chloride composite with a stabilizer content of 2 mass parts:
a – 1 cycle; *b* – 2 cycle; *c* – 3 cycle; *d* – 4 cycle. Red line – temperature, blue line – torque



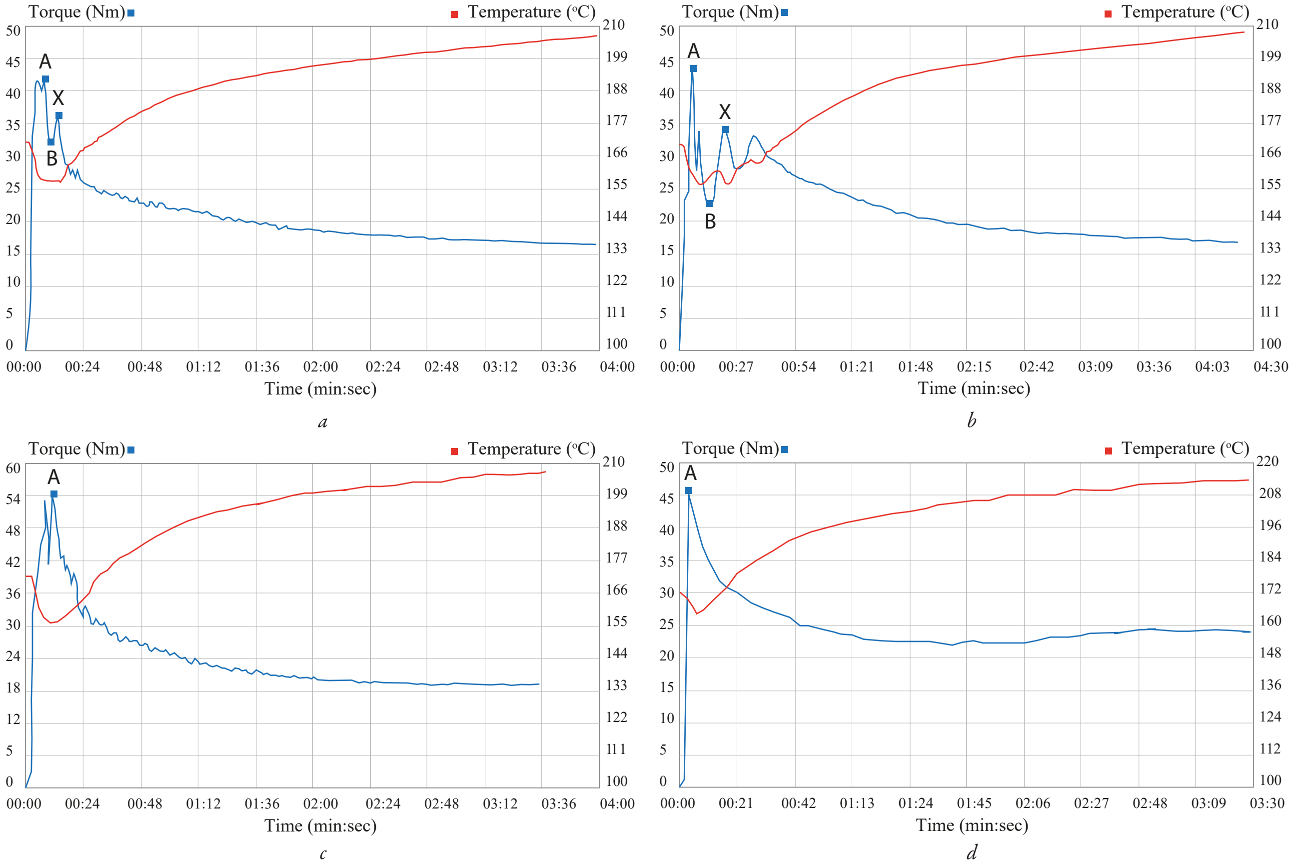
a

b

c

d

Fig. 3. Plastogram of the basic polyvinyl chloride composite with a stabilizer content of 3 mass parts: *a* – 1 cycle; *b* – 2 cycle; *c* – 3 cycle; *d* – 4 cycle. Red line – temperature, blue line – torque



a

b

c

d

Fig. 4. Plastogram of the basic polyvinyl chloride composite with a stabilizer content of 4 mass parts: *a* – 1 cycle; *b* – 2 cycle; *c* – 3 cycle; *d* – 4 cycle. Red line – temperature, blue line – torque

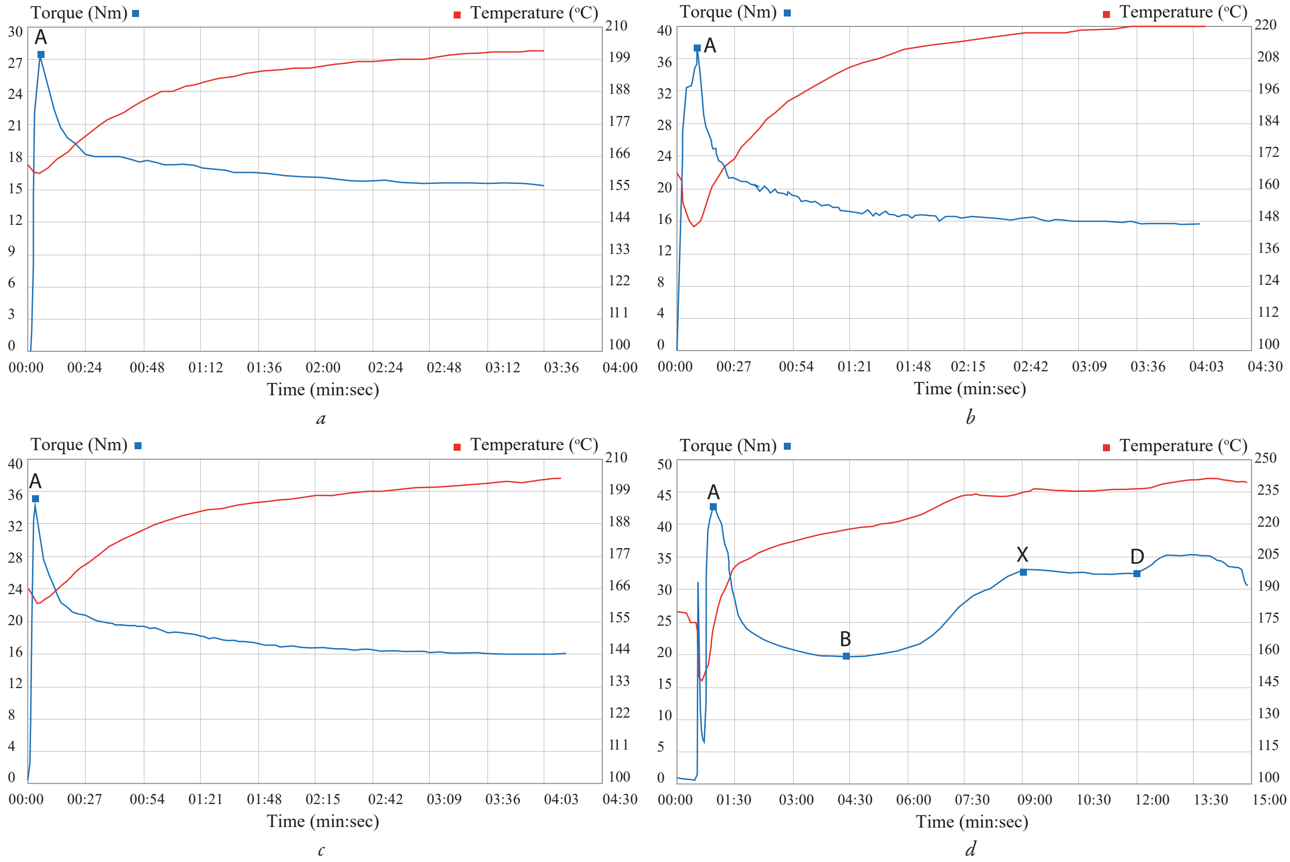


Fig. 5. Plastogram of the basic polyvinyl chloride composite with a stabilizer content of 5 mass parts: *a* – 1 cycle; *b* – 2 cycle; *c* – 3 cycle; *d* – 4 cycle. Red line – temperature, blue line – torque

Table 2

Physical and mechanical characteristics of the basic polyvinyl chloride composite with a stabilizer content of 4–5 mass parts depending on the recycling cycle

Processing cycle	1		2		3		4		5	
Stabilizer content, mass part	4	5	4	5	4	5	4	5	4	5
MFI, g/10 min	32	31	35	37	37	39	38	40	38	41
Tear strength, MPa	31	30	29	28	27	27	26	27	27	26
Elongation, %	19	18	17	17	16	14	15	13	15	12
Impact strength, kJ/m^2	27	28	36	35	39	40	37	38	36	36

Research limitations. To implement the research results, it is worth investigating the influence of other components of PVC formulations used in real production. It is also worth investigating other methods of physical modification of PVC and their combination.

The influence of martial law conditions. Interruptions in the power system of Ukraine affected the speed and quality of experimental research.

Prospects for further research. An important aspect is the study of other methods of physical modification of polyvinyl chloride, for example, the introduction of plasticizers or fillers. Plasticizers and fillers make it possible to facilitate the process of multiple recycling of PVC composites and modify their operational properties. Therefore, it is important to investigate the influence of these additives and determine their rational parameters.

4. Conclusions

It has been shown that there are significant opportunities for secondary processing of PVC materials and intensive work is being carried out to develop the processing of raw materials to meet the needs of a cyclical economy. A method of cyclic processing of PVC is proposed by introducing a rational amount of calcium/zinc heat stabilizer at the stage of primary processing. It has been established that increasing the content of heat stabilizer from 3 mass parts to 4–5 mass parts increase the life of PVC composite by 1.5–2 times. Such heat stabilization allows maintaining sufficiently stable physical and mechanical characteristics and molecular weight during 5 cycles of processing of PVC composite. Such an approach to PVC processing contributes to the implementation of the principles of sustainable development and is an important step towards the creation of closed technological cycles in industry.

Conflict of interest

The authors declare that they have no conflict of interest concerning this research, whether financial, personal, authorship or otherwise, that could affect the study and its results presented in this paper.

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Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirms that they did not use artificial intelligence technologies when creating the presented work.

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