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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS OF UNSATURATED CYCLIC (BIS)IMIDES
AND OBTAINING OF BIOLOGICALLY ACTIVE POLYMER
MATERIALS BASED ON THEM AND STUDY OF THEIR
PROPERTIES**

Speciality: 2306.01 – Organic chemistry
 2304.01 – Macromolecular chemistry
Field of science: Chemistry

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The work was performed at the laboratory "Polymeric materials based on macromonomers" at the Institute of Polymer Materials of Azerbaijan National Academy of Sciences

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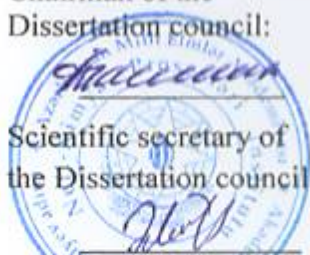
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GENERAL CHARACTERISTICS OF THE WORK

Actuality of the research and degree of elaboration. Synthesis of polyimides with a set of properties such as high thermal resistance¹, photosensitivity, optical activity, adhesion, resistance in a wide temperature range² (-80÷500) is one of the important directions of macromolecular chemistry. These properties allow wide use of polyimides in electronics, machinery, shipbuilding and aerospace. Chlorinated N-substituted bisimides of cyclic di- and tetracarboxylic acids are of particular importance. These types of imides can undergo step polymerization, radical copolymerization and various photochemical reactions due to unsaturated bonding, and allow the production of a wide range of polymeric materials.

Although there's a large amount of literature on production and application of unsaturated cyclic imides and halogenated derivatives³ of unsaturated dicarboxylic acids, there's a lack of sufficient data in literature on the synthesis of N, N'-bisimides of mixed diene-dienophilic fragments and their polychlorinated derivatives based on cyclic dicarboxylic acids and obtaining antibacterial polymer materials based on them.

As mentioned above, it should be noted that study of the methods for the synthesis of new unsaturated cyclic chlorinated N-substituted bisimides, polycondensation and copolymerization reactions based on bisimides, determination of their biological activity⁴ is one of the current issues of organic and macromolecular chemistry.

¹ Meador, M.B., Polyimides and process for preparing polyimides having thermal-oxidative stability, Pat 6274699, USA / Meador M.B. – 2001.

² Zhubanov, B.A., Polyimide as a heat-resistant, fire-resistant material. Pat. № 1066199, SSSR / B.A.Zhubanov, O.A. Almabekov, V.D. Kravtsova [et.al.] – 1982. № 25.

³ Vinogradova C.B. Polycondensation processes and polymers / S.V.Vinogradova, V.A.Vasnev - M.: Mir, - 2000, - 250 p.

⁴ Banilla, A.M., Polymeric materials with antimicrobial activity / A.M.Banilla, M.F.Garcia // Progress Polymer. Sci. – 2012, V.37. – p. 281-339.

The main object of the research. The main object of the research is the synthesis of polychlorinated bisimidodienes and determination of possibility for obtaining copolymers by cooligomerization reactions of the known spatial structured-polyimides with moving bodies and N,N'-(alkylene) and N,N'-(arylene) unsaturated cyclic bisimides with allyl(allylsalicylate) and vinyl(styrene) type monomers by Diels-Alder polycondensation reaction. But the subject of the research is the study of bactericidal and fungicidal activity of synthesized various bisimides, bisimidodienes, oligomers, copolymers.

The aims and objectives of the research. The aim of the research is developing effective methods for the synthesis of N, N'-R-bisimidodienes of polychlorinated cyclic dicarboxylic acids and bisimides with corresponding diene-dienophilic fragment, obtaining imide groups-containing polymeric materials, comparative study of the biological activity of the obtained compounds and determination of the application fields.

For the purpose of achieving the goal:

- Determination of optimal conditions for the synthesis of dianhydrido-, mono- and bisimidodienes of 2,3,4-tetrachlorocyclohexa-1,3-diene-5,6- and 2,3,4,5-tetrachlorobicyclo[4.4.0]deca-2,4-diene-8,9-dicarboxylic acids and diene-dienophile fragment-containing bisimides of polychlorinated cyclic dicarboxylic acids;
- Study of possibilities for obtaining polyimides based on bisimidodiene and bisdiene-dienophiles by Diels-Alder polycondensation reaction ;
- Determination of optimal conditions for the synthesis of polyimides by radical copolymerization of N, N'-(alkylene) and N,N'-(arylene) bisimides with allyl(allylsalicylate) and vinyl (styrene) type monomers;
- Obtainment of antibacterial polypropylene, polyethylene, acrylonitrile-butadiene-styrene and (polyphenylene oxide-containing) polycarbonate-based composite materials of "Noryl" using synthesized polychlorinated compounds and oligomers as biologically active additives;

- Study of practical application of biological activities of the composite materials based on the synthesized bisimides, including oligomers

Research methods. In carrying out this study was used modern physicochemical methods such as IR, ^1H ^{13}C NMR spectroscopies, methods of X-ray structural analysis, and thin-layer chromatography.

The crystal structures of the synthesized compounds were confirmed by the Bruker 6 APEX II CCD diffractometer. NMR ^1H and ^{13}C spectra were recorded on a Bruker Avance spectrometer in CDCl_3 and DMSO solvents. UT-254 was performed on NTX Silufol board, the formed spots were examined in UV lamp beams. Column chromatography was performed using Merck silicogel.

Antimicrobial activities of some synthesized compounds against various bacteria were studied by a serial dilution method.

The main provisions submitted to the defence

- Development of suitable methods for the synthesis of new unsaturated cyclic bisimides, mixed bisimidodienes and the results of the carried out reactions;
- Discussion of the results from X-ray study of structural properties of the synthesized new compounds –2,3,4,5,7,7-hexachlorobicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid N,N'-(1,6-hexyl)bisimidine monocystal;
- Obtainment of polyimides based on bisimidodiene and mixed bisdiene-dienophiles by Diels-Alder polycondensation reaction;
- The results and discussion of the study on cooligomerization reactions of N,N'-(alkylene) and N,N'-(arylene) unsaturated cyclic bisimides with allyl (allylsalicylate) and vinyl (styrene) monomers;
- The results of the study on antibacterial and antifungal activities of the synthesized compounds.

Scientific novelty of the research. For the first time, diene-dienophilic bisimides of polychlorinated cyclic dicarboxylic acids were synthesized efficiently with high yield, diene activity of diene synthesis reaction of obtained bisimidodienes and maleic anhydride was studied and the ways for regiocontrolled synthesis of

bisimidodianhydrides combined with polychlorinated bicyclooctene or cyclohexane fragment of anhydride rings were proposed.

Polyimides were synthesized from diene-dienophilic bisimides unknown in the literature via Diels-Alder polycondensation reaction and optimal conditions were determined for the reactions. Regularities of cooligomerization reactions of N, N'-(alkylene) and N,N'-(arylene) unsaturated cyclic bisimides with allyl (allylsalicylate) and vinyl (styrene) monomers were studied and it was shown that the reactions occurs due to the formation of donor-acceptor complexes by charge-transfer between monomer pairs and sequence-defined macromolecules are formed.

The study of biological activities of synthesized polychlorinated compounds and imide-containing oligomers revealed that most of these compounds possess antibacterial and antifungal properties. It was noted that some of them may be used as additives in the production of antibacterial polyethylene, polypropylene, acrylonitrile-butadiene-styrene and "Noryl" polyoxyphenylene-based composite materials. It was found that adding 0.1-1.0 wt% of these antibacterial additives to the above-mentioned composition materials during the extrusion (molding) stage causes formation of higher bactericidal and fungicidal properties of these polymeric materials while maintaining physical and mechanical properties.

Theoretical and practical significance of the research. Synthesis of bisimidodienes, cyclic di- and tetracarbon acids and diene-dienophilic bisimides on the basis of their halogenated derivatives by a more convenient and simple way, results from their copolymerization and polycondensation reactions enriched theoretical knowledge in organic and polymer chemistry, PE, PP, ABS and "Noryl" polycarbonate-based composite materials were obtained using the oligomers based on diene-dienophilic bisimides of polychlorinated cyclic dicarboxylic acids and unsaturated cyclic bisimides as biologically active additives.

The synthesized compounds may be used as antibacterial polymeric materials in agriculture, medicine-; modifiers for obtaining adhesive composition for optical devices; semi-volcanic retarders-; adhesion additives to the adhesive composition for

photoreceptors-; antipyrene for obtaining refractory materials from medium pressure polyethylene and ED-20 epoxy resin.

Approbation and study of the research. 39 scientific works, including 15 papers, 23 abstracts have been published and 1 patent have been obtained on the materials of the dissertation. The results have been presented and discussed in the following conferences:

Scientific conferences of postgraduate students of ANAS (Baku, 2005, 2006 və 2007), VI Baku International Y.Mamedaliyev petrochemistry conference, (Baku, 2005), III International Scientific Conference on "Fine Organic Synthesis and Catalysis" dedicated to the 85th anniversary of the ASOA (Baku, 2005), II Republican scientific conference "Young chemist" (Baku, 2006), Scientific conference dedicated to the 100th anniversary of Academician H. Afandiyev (Baku, 2007), Scientific conference dedicated to the 100th anniversary of Academician M.F. Nagiyev (Baku, 2008), The Republican conference "Organic Reagents in Analytical Chemistry" dedicated to professor A.A. Verdizada's 95th anniversary of birth (Baku, 2009), Republican scientific conference "Additives and reagents that improve the quality of materials and protect them", dedicated to the 100th anniversary of Prof. A.M. Guliev. (Baku, 2012), II Republican scientific conference on "Modern problems of chemistry of monomers and polymers" dedicated to the 50th anniversary of SSU (Sumgayit, 2012), Republican scientific conference dedicated to academician A.A. Afandiyev's 75th anniversary of birth (Sumgayit, 2013); Republican scientific conference "Macromolecules chemistry, organic synthesis and composite materials" dedicated to the 50th anniversary of the Institute of Polymer Materials (Sumgayit, 2016), Integration processes of the world science in the 21st century (Ganja, 2016), International scientific-technical conference on "Petrochemical synthesis and catalysis in complex condensed systems" dedicated to the 100th anniversary of B.G. Zeynalov (Baku, 2017), Scientific conference "Naghiyev Readings" dedicated to academician M.Naghiyev's 110th anniversary of birth (Baku, 2018), I National Students Scientific Conferences dedicated to the 96th anniversary of the National leader of Azerbaijan Heydar Aliyev, (Baku, 2019), The

International Scientific Conference “Actual of Modern Chemistry” dedicated to the 90th anniversary of the Academician Y.H.Mammadaliyev Institute of Petrochemical Processes, (Baku, 2019), Innovative development perspectives of chemical technology and engineering international scientific conference, (Baku, 2019), I International Student Research and Scientific Conference of on "Sustainable Development in Chemistry and Chemical Engineering" Dedicated to the 97th Anniversary of the National Leader Heydar Aliyev, (Baku, 2020), IV All-Russian Conference "Chemistry and Chemical Technology: Achievements and Prospects", (Moscow, 2020), The VIII International Scientific Conference «The chemistry of coordination compounds» dedicated to the 85th anniversary of the Department of Analytical Chemistry (Baku, 2020).

The name of the organization where the dissertation work was performed. The dissertation work was performed at the laboratory of “Polymeric materials based on macromonomers” on the research plan of the Institute of Polymer Materials of ANAS (State registration number 0106Az0114).

The applicant’s personal presence. The main goals of the research and the ways for solving target issues were revealed, the directions of the researches were determined, the results were discussed by the applicant. Simultaneously, the applicant directly participated in carrying out laboratory experiments and each stage of writing papers, as well as the dissertation.

The scope and structure of the dissertation. The dissertation consists of 204 pages including: introduction – 7 pages (13.000 symbols), four chapters, literature review – 40 pages (42.087 symbols), discussion of the researches (the second chapter) – 40 pages (37.203 symbols), synthesis and applications of polymers (the third chapter) – 46 pages (48.166 symbols), experimental methods (the fourth chapter) – 36 pages (48.205 symbols), results – 3 pages (3.765 symbols), 225 references – 26 pages, 53 figures and 25 tables, and volume – 192426 symbols (with the exception of figures, tables, references and appendices).

Introduction describes the actuality, objectives, scientific novelty, practical significance, approbation, structure and scope of the topic, publications on it, essence of the chapters briefly.

The first chapter deals with the analysis of recent literature data on the discussion of the synthesis, conversion, and determination of the biological activity of polychlorinated polycyclic dicarboxylic acid bisimides, as well as the discussion of bisdienes with bismaleinimides and diene-dienophilic bisimides by Diels-Alder reaction.

The second chapter discusses the methods for effective synthesis of polychlorinated polycyclic unsaturated dicarboxylic acids N, N'-R-bisimides and diene-dienophilic bisimidodienes, the results of the study on the composition and structure of the synthesized compounds. Simultaneously, for the first time, it was described the synthesis of bisimidodiene, containing both a diene and a dienophile fragments in the molecule based on bisimides of these acids and maleic anhydride.

The third chapter deals with the results of Diels-Alder polycondensation reaction of N, N'-R-bisimides of unsaturated dicarboxylic acids and diene-dienophilic bisimidodienes, as well as obtaining antibacterial oligomers by radical copolymerization with vinyl monomers. Simultaneously, there were presented the methods for obtaining the composite materials based on PE, PP, ABS and "Noryl" polycarbonate with these antibacterial bisimides and oligomers. Analyzing of application fields of some of the synthesized bisimides, their antibacterial and antifungal properties were also given in this chapter.

The fourth chapter is experimental part of the work. Physicochemical properties of the primary substances and synthesized compounds are also set in this chapter.

The Results section reflects the major findings of the research.

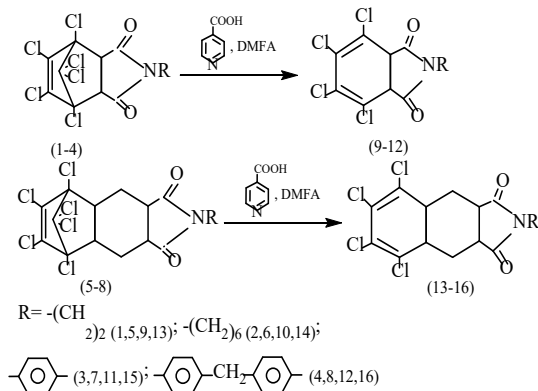
THE MAIN CONTENT OF THE WORK

1. Synthesis of N, N'-bisimides of 1,2,3,4-tetrachlorocyclohexa-1,3-diene 5,6- and 2,3,4,5-tetrachlorobicyclo[4.4.0]deca-2,4-diene 8,9-dicarboxylic acids

Chlorinated bisimidodienes of bi- and tricyclic dibasic acids are promising monomers for obtaining chlorine-based refractory and thermo-resistant polyimides.

Before synthesizing of N, N'-bisimidodienes, 1,2,3,4-tetrachlorocyclohexa-1,3-diene 5,6- (9-12) and 2,3,4,5-tetrachlorobicyclo[4.4.0]deca-2,4-diene 8,9-dicarboxylic acids (13-16) imidodienes were obtained on the basis of known spatial structured 2,3,4,5,7,7-hexachlorobicyclo[2.2.1]hept-5-ene-2,3- (HCBCHDA) (1-4) and 1,2,3,4,11,11-hexachlorotricyclo-[6.2.1.0^{5,10}]undec-2-ene-7,8-dicarboxylic acids (HCTCUDA) (5-8) imides.

A catalyst-acceptor - isonicotinic acid is added to solutions of HCBCHDA (1-4) or HCTCUDA (5-8) imides via regular stirring at room temperature under dimethylformamide medium. The reaction is exothermic and temperature rises up to 50°C. The reaction mixture is reheated at 120÷140°C for another 2 h.



Effect of temperature, reaction time, molar ratios of components on the yields of adducts (9, 13) was studied. Optimal conditions for the synthesis of imidodiene (9) were determined as: temperature 140°C, time - 6 h and the mol ratio of diene-dienophile - 2:1. Synthesis of imidodiene (13) was carried out under the following

optimal conditions: temperatur 150°C, time – 8 h and mol ratio of diene-dienophile – 3:1. The yield of imidodienes (9, 13) was 90-95 %.

The structure of the synthesized imidodienes (9-16) has been studied by IR, UV, ¹H NMR and ¹³C NMR spectroscopies, methods of X-ray structural analysis, but the purities have been determined by thin layer chromatography (TLC) on the plate of “Silufol UV-254” in the system of corresponding solvents.

Absorption bands in 3280 and 3420 (NH₂), 1710 and 1780 (C=O), 1603-1610 (C=C) and 630-750 cm⁻¹ (C–Cl) have been assigned to the corresponding fragments in IR spectrum of imidodienes (9-16).

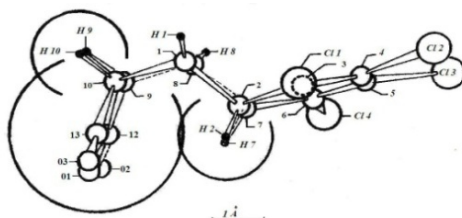
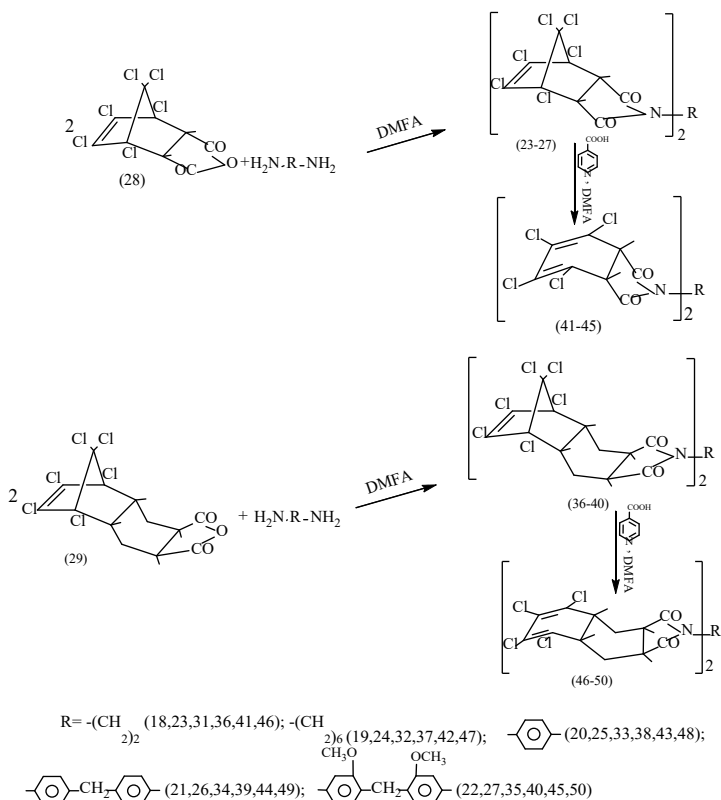


Fig. 1. Molecular structure of N-(1,2-ethane) imidodiene endo-, exo-2,3,4,5-tetrachlorobicyclo[4.4.0]deca-2,4-diene-8,9-dicarboxylic acid

The maximum is assigned at 285–287 nm corresponding to π - π^* transition and confirming the presence of a diene chromophore characteristic for soid homoannular diene systems in the UV spectrum of imidodienes.

For obtaining polyimides of moving body, HCBCHDA (23-27) and HCTCUDA (36-40) bisimides were synthesized acylation of 2,3,4,5,7,7-hexachlorobicyclo[2.2.1]hept-5-en-2,3-dicarboxylic acid (HCBCHDA) or 1,2,3,4,11,11-hexachlorotricyclo[6.2.1.0^{5,10}]undec-2-en-7,8-dicarboxylic acid (HCTCUDA) anhydrides with diamines.

Planar structure bisimidodienes (41-45 and 46-50) are obtained by the reaction of bisimides (34-43) under DMFA medium. Cross synthesis of diacylation of anhydridodienes with diamines was carried out according to the following scheme to confirm the structure of bisimides (41-45 and 46-50).



Effect of temperature, reaction time, molar ratios of components on the yields of bisimides (23-27) and (36-40) was studied. Optimal conditions for the synthesis of bisimides (23, 25) were determined as: temperature 120°C, time - 8 h and the mol ratio of diene-dienophile - 2:1. Synthesis of bisimides (36, 40) was carried out under the following optimal conditions: temperatur 140°C, time – 10-12 h and mol ratio of diene-dienophile – 3:1. The yield of bisimides (23, 25) and (36, 40) was 89-92 %.

The structure of the synthesized bisimides (23-27) and (36-40) has been studied by IR and methods of X-ray structural analysis.

Absorption band of –NH– and NH₂ fragments disappears which has been observed in 3280, 3350 cm⁻¹ in IR spectrum of bisimides and absorption band appears in the region of 1720-1780 cm⁻¹ that reveals the presence of carbonyl group (C=O) characteristic for the

synthesized bisimides (23-27) and (36-40). Absorption bands have been assigned in the frequency range 630-750 cm^{-1} for C–Cl communication.

Absorption bands in 1710 and 1780 ($\text{C}=\text{O}$), 1602-1610 ($\text{C}=\text{C}$) and 630-750 cm^{-1} ($\text{C}-\text{Cl}$) have been assigned to the corresponding fragments in IR spectrum of bisimidodienes (41-50).

2. Stereochemistry of N,N'-R-bisimides of 2,3,4,5,7,7-hexachlorobicyclo[2.2.1]hept-5-ene-2,3-, 1,2,3,4,11,11-hexachlorotricyclo[6.2.1.0^{5,10}]undes-2-ene-7,8-, 1,2,3,4-tetrachlorocycloheksa-1,3-diene-5,6- v α 2,3,4,5-tetrachlorobicyclo[4.4.0]deka-2,4-diene-8,9-dicarboxylic acids

Intermolecular halogen bonds and other types of non-covalent interactions in halogenated N-heterocyclic compounds can improve their solubility and other functional properties. In this direction, new halogenated N-heterocyclic compounds with a large number of non-covalent intermolecular interactions have been synthesized.

From the synthesized compounds 2,3,4,5,7,7-hexachlorocyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid N, N'-(1,6-hexyl)bisimide (24) were obtained single crystals whose molecular structure was confirmed by methods of X-ray structural analysis.

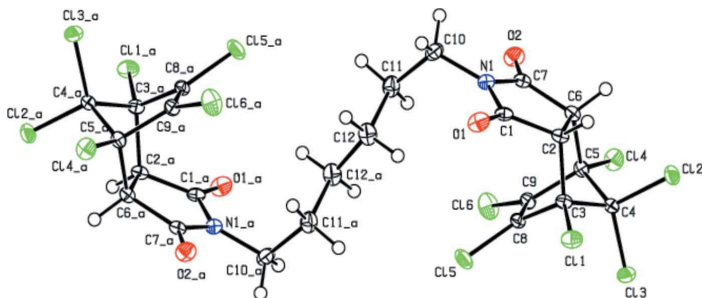


Fig. 2. The molecular structure of 2,3,4,5,7,7-hexachlorocyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid N, N'-(1,6-hexyl)bisimide

The molecule of the title compound (24) is generated by a crystallographic inversion centre at the midpoint of the central C–C bond. A kink in the molecule is defined by the C10-C11-C12-C12_o a torsion angle of 169.86 (15) $^\circ$ about this central bond of the alkyl bridge (Fig. 2). The pyrrolidine ring (N1/C1/C2/C6/C7) is essentially

planar [maximum deviation=0.014(1)Å]. The cyclohexane ring (C2/C3/C5/C6/C8/C9) has a boat conformation [the puckering parameters $Q_T = 0.9300$ (14) Å, $\theta = 89.99$ (9)°, $\varphi = 59.37$ (9)°], while both the cyclopentane rings (C2-C6 v \grave{a} C3-C5/C8/C9) adopt an envelope conformation [$Q(2) = 0.6308$ (14) Å, $\varphi(2) = 252.44$ (13)° v \grave{a} $Q(2) = 0.5835$ (14) Å, $\varphi(2) = 215.53$ (14)°, respectively] with the C4 atom bearing the dichloromethane group as the flap.

Hirshfeld surface analysis for the 2,3,4,5,7,7-hexachloro-cyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid N,N'-(1,6-hexyl)-bisimide. In the crystal structure, molecules are linked by intermolecular C–H \cdots O, C–H \cdots Cl and C–Cl \cdots interactions (Table 1), and short intermolecular contacts, forming a three-dimensional network.

In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface analysis was carried out using *Crystal Explorer* 17.5. Fig. 3 shows the Hirshfeld surface plotted over d_{norm} in the range -0.1922 to 1.7149 a.u. The red spots on the Hirshfeld surface represent C–H \cdots O and C–H \cdots Cl contacts. The full two-dimensional fingerprint plot and those delineated into the major contacts: Cl \cdots H/H \cdots Cl (33.6%), Cl \cdots Cl (29.3%), O \cdots H/H \cdots O (13.9%), Cl \cdots O/O \cdots Cl (11.4%) and H \cdots H (7.0%) interactions. The remaining other weak interactions (contribution percentages) are Cl \cdots C/C \cdots Cl (3.2%), Cl \cdots N/N \cdots Cl (1.4%) and C \cdots H/H \cdots C (0.2%).

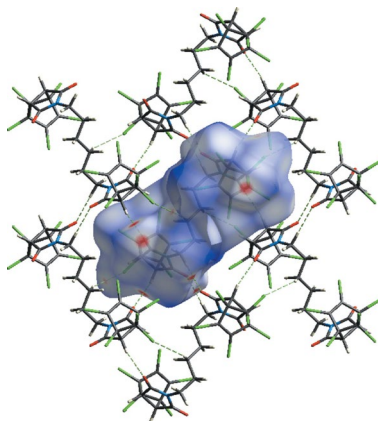


Fig. 3. A view of the Hirshfeld surface for the compound (24)

The pyrrolidine ring makes a dihedral angle of 14.83 (12)_° with the 3,4-dimethoxyphenyl ring, which are attached to each other by an extended N-CH₂-CH₂-C_{ar} bridge. The molecules are stacked in layers held together by offset π-π interactions, with a centroid-centroid distance of 3.564 (1) Å for the pyrrolidine and benzene rings. There is also an intermolecular C—Cl⋯ interaction present.

Table 1. Hydrogen-bond geometry (Å).

D—H⋯A	D—H	H⋯A	D⋯A	DH⋯A
C6—H6⋯O1 ⁱ	1.00	2.43	3.3867 (16)	161
C10—H10A⋯O2 ⁱⁱ	0.99	2.45	3.4402 (17)	178
C12—H12B⋯Cl2 ⁱⁱⁱ	0.99	2.80	3.5299 (15)	131
C3—Cl1⋯Cg1 ⁱⁱⁱ	1.75 (1)	3.89 (1)	4.9389 (14)	117 (1)

The structure of the synthesized bisimid (24) has been studied by IR, ¹H NMR and ¹³C NMR spectroscopies. ¹H NMR (300.130 MHz) in acetone-d₆, internal TMS, δ (ppm): 1.29–3.43 multiplet, triplet (12H, 6CH₂), 3.86 singlet (4H, CH). ¹³C {¹H} NMR (75.468 MHz, acetone-d₆). δ: 25.8 (2CH₂), 27.2 (2CH₂), 39.3 (4C—H), 52.0 (2CH₂), 79.3 (4CCl), 104.4 (2CCl₂), 130.9 (2ClC=CCl) and 170.2 (4C=O). Off-white prismatic crystals suitable for X-ray analysis were obtained by slow evaporation of a chloroform–hexane (1/1, v/v) mixture. (Fig. 4).

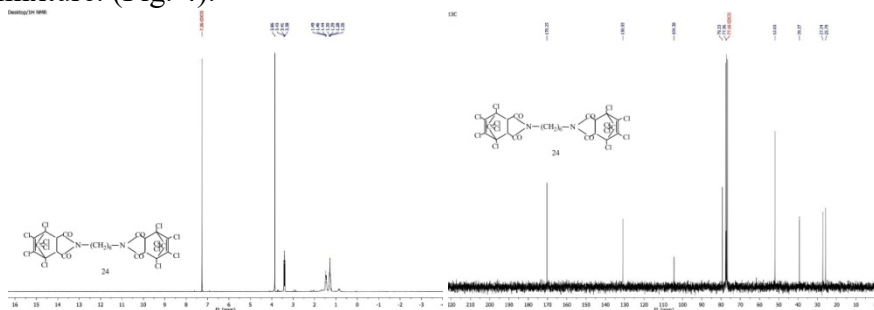


Fig 4. ¹H and ¹³C NMR spectrum of N, N'-(1,6-hexyl)bisimide of 2,3,4,5,7,7-hexachlorocyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid

Also, ¹H and ¹³C NMR spectrum of N, N'-(4,4'-diphenylmethane) bisimide of 1,2,3,4,11,11-hexachlorotricyclo-[6.2.1.0^{5,10}]undes-2-ene-7,8-dicarboxylic acid (39) presents ¹H NMR

m.h.: 1.43 (triplet, CH), 2.73 (CH₂), 3.91 (CH₂), 4.07 (CH), 7.00-7.33 (CH, Ar.) signals, ¹³C NMR have been observed in the regions of 21.25 (CH₂), 37.62 (CH), 53.47 (CH₂), 81.92 (Cl-C-C), 102.41 (Cl-C-Cl), 126.15, 126.39, 128.35, 129.72 (C, Ar.), 131.59 (C-Cl), 177.73 (C=O).

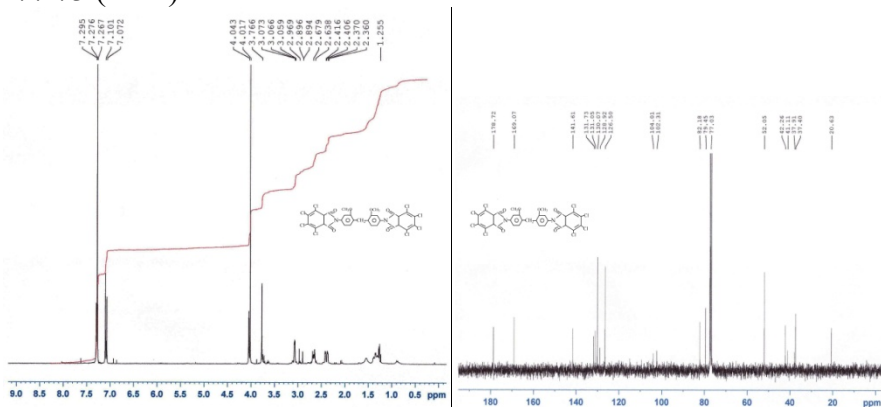
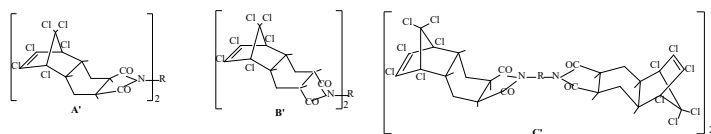


Fig 5. ¹H and ¹³C NMR spectrum of N,N'-(3,3'-dimethoxy-4,4'-diphenylmethane) bisimide of 1,2,3,4-tetrachlorocyclohexa-1,3-diene-5,6-dicarboxylic acid (45)

The structure of the synthesized of N,N'-(3,3'-dimethoxy-4,4'-diphenylmethane)bisimide of 1,2,3,4-tetrachlorocyclohexa-1,3-diene-5,6-dicarboxylic acid (45) has been studied by ¹H and ¹³C NMR spectroscopies. ¹H NMR m.h.: 3.76 (singlet, 4H, CH), 4.01 (singlet, 6H, CH₃), 4.04 (singlet, 2H, CH₂), 7.07, 7.10, 7.29 (6H, CH, Ar.) signals, ¹³C NMR have been observed in the regions of 20.63 (CH), 37.40 (CH₂), 52.05 (OCH₃), 79.45 (Cl-C-C), 104.01 (Cl-C-Cl), 126.50, 128.92, , 131.05, 131.73, 141.61 (C, Ar.), 130.07 (C-Cl), 169.07 (C-O), 178.72 (C=O).

It is known that anhydride and N-phenylimide of *cis*-4-THDA are in *syn*- and *anti*-boat conformational states at high temperatures. Both states are equally probable to form an *endo*-oriented complex, and the corresponding adduct with *endo*, *exo-endo*, *exo*- (A), *endo*, *endo-endo*, *endo*- (B) and *endo*, *exo-endo*, *endo*-imide (C) residual configuration can be obtained. Condensation reaction occurs at high temperatures (150°C), therefore N, N'-bisimides of *cis*-4-THDA (36-40) can interact in three conformational states (A-C):

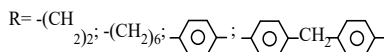
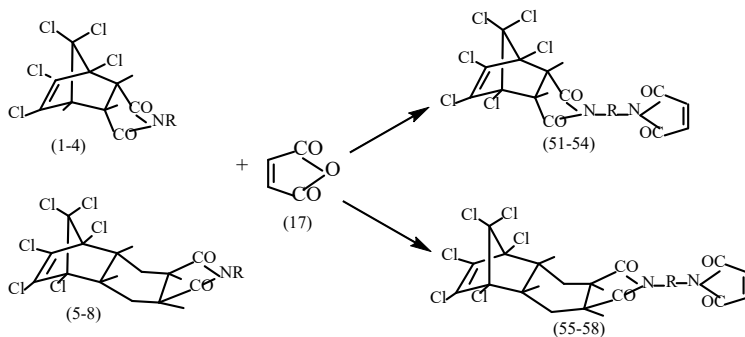


Specific signals of methylene protons are assigned at 3.42-3.50 ppm in the NMR spectrum of the obtained bisimides (36-40). As a result of double cyclic substitution in cyclohexane ring, a boat conformation is possible, ensuring the coplanarity of the bonds on the 5, 10 and 7, 8th carbon atoms.

3. Synthesis of diene-dienophile fragment-containing bisimides of polychlorinated di- and tricyclic dicarboxylic acids

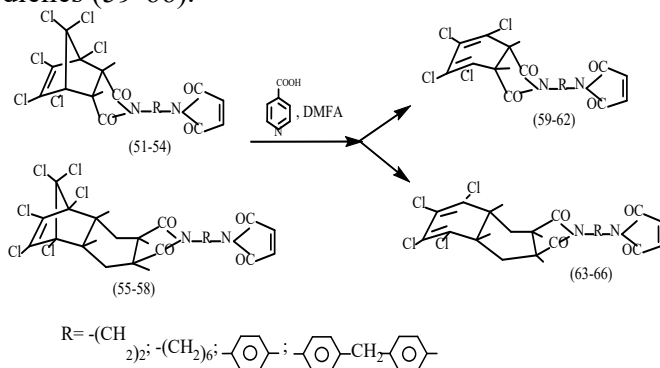
In order to increase the range of polychlorinated cyclic dienes and to further improve the technology of obtaining bisimidodienes, mixed bisimidodiene-dienophiles with both diene and dienophil fragments in one molecule were synthesized.

N-imides of HCBCHDA (1-4) v \grave{a} HCTCUDA (5-8) with maleic anhydride at a temperature of 140 $^{\circ}$ C with a 1: 1 mol ratio of the components in a DMFA environment, HCBCHDA (51-54) and HCTCUDA N, N'-maleininimidoalkyl(aryl) substitute images (55-58) were obtained.



Absorption bands in 1780 (C=O), 1590-1600 (C=C) and 650-780 cm^{-1} (C-Cl) have been assigned to the corresponding fragments in IR spectrum of bisimides (51-58).

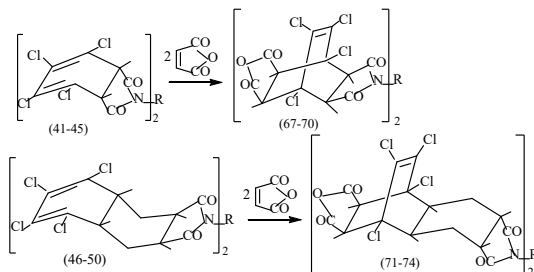
A catalyst-acceptor - isonicotinic acid is added to solutions of N, N'-maleinimidoalkyl (aryl) bisimides (51-58) via regular stirring at room temperature under dimethylformamide medium. The reaction is exothermic and temperature rises up to 50°C. The reaction mixture is reheated at 120÷140°C for another 2 h and obtained suitable bisimidodienes (59-66).

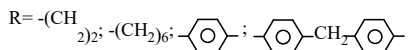


The structure of the synthesized bisimides (59-66) has been studied by IR, UV, ^1H NMR and ^{13}C NMR spectroscopies. Absorption bands in 1720 and 1780 (C=O), 1603-1610 (C=C) and 660-780 cm^{-1} (C-Cl) have been assigned to the corresponding fragments in IR spectrum of bisimide (63).

4. Synthesis of N,N'- bisimidodianhydrides chlorinated di- and tricyclic tetracarboxylic acids

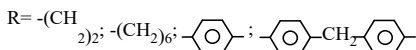
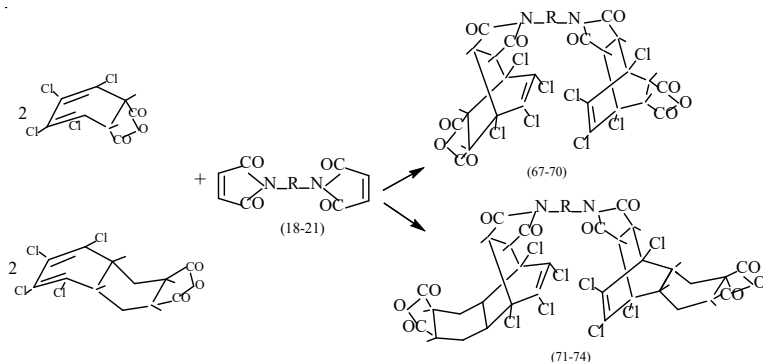
Studies have shown that N, N'-bisimidodiene (41-45) and (46-50) easily react with diene condensation with maleic anhydride:





Absorption bands in 1840 and 1865 (C=O), 1600-1610 (C=C) and 660-780 cm^{-1} (C-Cl) have been assigned to the corresponding fragments in IR spectrum of bisimidodianhydrides (67-74).

Bisimidodianhydrides (67-74) can also be obtained by cross synthesis - a single-step Diels-Alder polycondensation reaction of anhydrides with bismaleinimides.



Based on the Stuart-Briegleb model and determination of dipole moments ($\mu_{\text{cal.}} - 3.87 \text{ D}$, $\mu_{\text{exp.}} - 3.80 \text{ D}$), it is evident that bisimidodianhydrides (71-74) are *endo*, *exo-endo*, *exo-*, but bisimidodianhydrides (67-70) possess *endo*, *endo-endo*, *endo*-configuration due to the rigid structure of anhydridodiene.

Effect of temperature, reaction time, molar ratios of components on the yields of bisimidodianhydrides (67-74) was studied. Optimal conditions for the synthesis of bisimidodianhydrides (67, 69) were determined as: temperature 150°C, time - 8 h and the mol ratio of diene-dienophile - 2:1. The yield of bisimidodianhydrides (67-74) was 91-94 %. An increase in temperature and subsequent increase in reaction time lead to the formation of resin-like by-products.

If we look through *endo*-direction of addends, possibility of approaching diene system of maleic anhydride planar molecule by *syn*- or *anti*-sides, bisimidodianhydrides (71-74) can be in four possible stereoisomeric structures - *endo*, *exo*-, *endo*, *endo*-, *exo*,

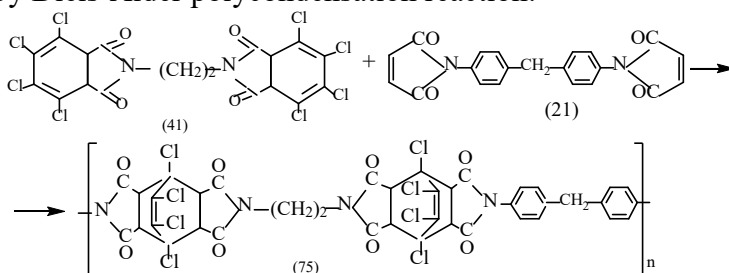
endo-, *exo*-, *exo*-. However, in practice, there is only one boat configuration in the cyclohexane ring - *endo*, *exo-endo*, *exo-stereoisomer*.

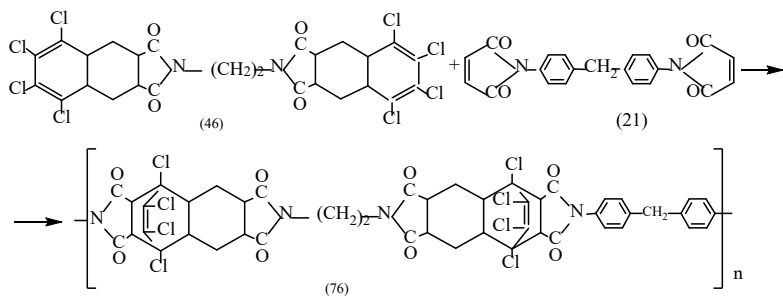
5. Study of antibacterial properties of synthesized cyclic bisimide derivatives

The effect of the composition and structure of some synthesized cyclic bisimides on the antibacterial properties was studied. It was determined that *endo*, *exo*-1,2,3,4,11,11-hexachlorotricyclo [6.2.1.0^{5,10}]undes-2-ene-7,8-dicarboxylic acid N-[2-maleinimido]ethyl bisimide (8) and N-[4-maleinimidobenzyl] phenyl bisimide of 1,4,5,6,7,7-hexachlorocyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid (58)) have more active antimicrobial and fungicidal properties.

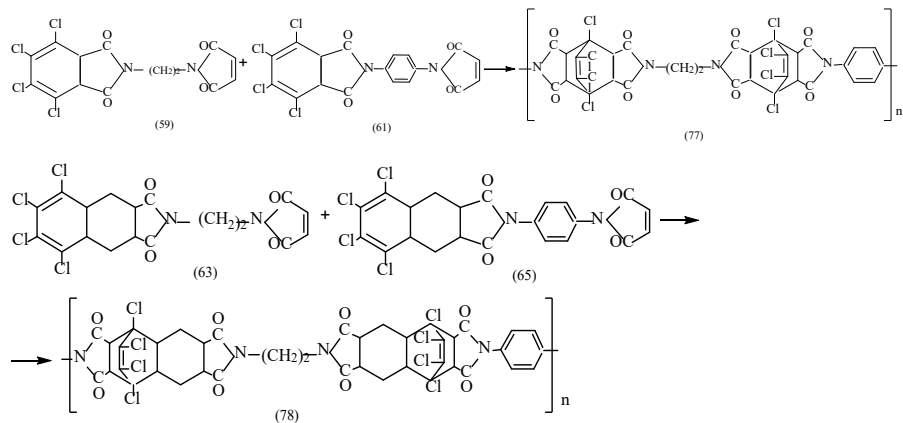
6. Diels-Alder polycondensation reaction of N, N'-(alkilen)-bisimides of 1,2,3,4-tetrachlorocyclohexa-1,3-diene 5,6- and 2,3,4,5-tetrachlorobicyclo[4.4.0]deca-2,4-diene 8,9-dicarboxylic acids with bismaleinimides and bisdiene-dienophiles bisimidodienes

In order to show possible synthesis of well-known hinge-ladder polyimides with the desired spatial structure was performed TCCHDDA (41) and TCBCDDDA with N, N'-(1,2-ethane) bisimidodienes (46) and N,N'-(4,4'-diphenylmethane) bismaleinimide (21) by Diels-Alder polycondensation reaction:





It has been shown that, it is possible to obtain polychlorinated, hinge-ladder polyimides from diene-dienophile fragmented bisimides. For this purpose, bisimides of TCCHDDA- (59, 61) and endo, exo-TCBCDDDA (63, 65) were selected and polycondensation of Diels-Alder reaction was carried out in DMAA environment:



In the process of polycondensation by the Diels-Alder the choice of the solvent in solution plays a decisive role. We have studied several aprotic solvents (DMF, DMA and DMSO). It was found that with increasing of the solvent polarity the viscosity and the yield of polyimide is increased. The greatest yield of polymer is reached when using DMAA as solvent. It reaches 90-95 %.

To determine the optimal conditions for the synthesis of polyimides (75, 76, 77, 78), the influence of the concentration of the initial monomer, the temperature, and the duration of the reaction on the course of polycondensation of bisimides has been studied (Fig. 6).

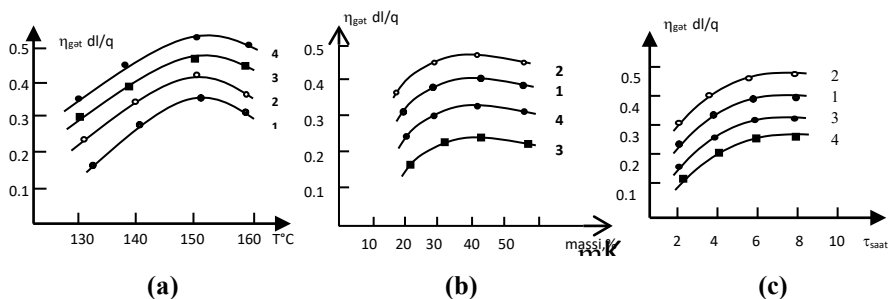


Fig. 6. Dependence of η_{iv} on of duration, on temperature of 0.5% solution of polyimides

The optimum monomer concentration is – 20-25 mass,%. Increasing the concentration above the optimum leads to a decrease in the MM polyimide. The reaction was carried out at 150°C and a duration of 6–8 hours. $\eta_{re}=0.4-0.5$ l/g. In the IR spectrum of polyimides (75 and 78) there are the characteristic absorption bands the bield of 1712, 1780 cm^{-1} (in the doublet 1720 cm^{-1}) for the C=O, 1590-1610 cm^{-1} for the C=C, 600–750 cm^{-1} corresponding for the C–Cl bonds vibrations.

The obtained PI (75, 76, 77, 78) are well soluble in polar solvents (DMF, DMAA, N-methylpyrrolidone, DMSO and pyridine). They are heat-resistant up to 390-400°C. The loss in weight PI of this temperature is 5-6%.

7. Study of copolymerization reactions of unsaturated cyclic bisimides

Studies have shown that cyclic bisimides containing diene-dienophil groups are easily converted to oligomers by polycondensation reactions by the Dils-Alder mechanism.

There is sufficient literature data on polymerization reactions of unsaturated cyclic bisimide derivatives with donor-type vinyl monomers. Sequence-structure oligomers are obtained as a result of these reactions. Many of cyclic bisimides and oligomers derived from them are biologically active compounds. Poor water-solubility and high temperature resistance allow these compounds to be used as biologically active additives in the production of antibacterial composite materials.

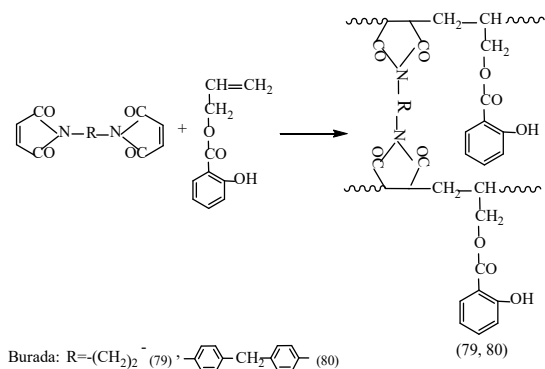
7.1. Obtaining and study of copolymerization of maleic bisimides with allyl ether of salicylic acid

The purpose of this section of the dissertation is to study the copolymerization reactions of cyclic (bis)imides of maleic acid with allyl ester of salicylic acid to obtain polymers containing cyclic bisimide group.

The purpose of selecting Allsat as a comonomer is to obtain polyfunctional copolymers containing both phenol and imide groups and to study their antibacterial and antifungal properties. Formation of a donor (D)-acceptor (A) type complex (a complex formed by charge-transfer) between the molecules of Allsat and bismaleinimide is known in the literature.

Considering this, NMR spectra of monomers were recorded separately and in mixtures of different proportions. The change in the value of chemical displacement of protons belonging to the BMI molecule in the NMR spectrum of a mixture of different compositions of phenyl bismaleinimide and Allsat monomers ($A \gg D$) indicates formation of a complex of type $[D \dots A]$.

The change in the value of this shift increases with molar ratio of the donor in monomer mixture. Equilibrium constant (K_c) of the complex formation process was calculated graphically using the Ketelaar equation. According to the calculations, $K_c = 0.097$ for the Allsat-Phenyl bismaleinimide monomer pair and $K_c = 0.082$ for the Allsat-Ethyl bismaleinimide monomer pair.



Naturally, the weak complex between the monomers has a significant effect on their relative reactivity. $r_1 = 0.05$ and $r_2 = 0.04$ for Allsat-Ethyl bismaleinimide monomer pair and $r_1 = 0.02$ and $r_2 = 0.05$ for the Allsat-Phenyl bismaleinimide monomer pair.

The values of relative activities of monomers in copolymerization reactions and their yields are close to zero that proves alternative (sequential) structure of copolymers.

Table 2.

Allsat and EBMI; Allsat and FBMI; St and EBMI; St and FBMI
Relative activity of monomers in radical copolymerization reactions of St and FBMI monomer pairs: ($t = 65^\circ\text{C}$, solvent dioxane, initiator-benzoyl peroxide (0.2%), reaction time -8 hours).

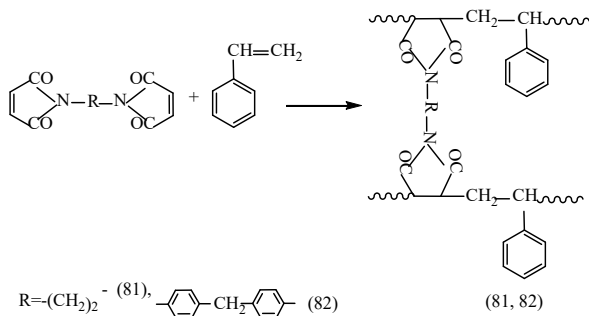
Monomer pairs		Copolymerization constants	
M ₁	M ₂	r ₁	r ₂
Al _{st}	EBMI	0.5	0.04
Al _{st}	FBMI	0.2	0.05
St	EBMI	18.2	0.02
St	FBMI	12.5	0.01

The antibacterial properties of Allsat – EBMI and Allsat - FBMI copolymers have been studied and it has been determined that the obtained polymeric materials have antibacterial and antifungal properties.

7.2. Obtaining and study of copolymerization of maleic bisimides with styrene

It is known that one of the wide application fields of PS is production of packaging products. It is used to make various packaging products for storing fruit, eggs, meat and fish products by thermoforming methods. One of the important conditions is their resistance to many bacteria and microfungi.

This section of the dissertation is devoted to the development of technology for the synthesis of styrene polymers with unsaturated malein bisimides for the production of antibacterial polystyrene (PS) and the study of their physico-mechanical and antibacterial properties.



Copolymers containing different amounts of BMI elemental composition unit in styrene were synthesized, and relative activities of monomers (r_1 and r_2) in copolymerization reactions were determined, dependencies such as composition-composition, composition-properties were determined. Comparison of the IR spectra of polystyrene and styrene copolymers with BMIs reveals that, unlike IR spectra of polystyrene, absorption bands of the ether group of the compound are assigned at 1694 cm^{-1} of IR spectra of copolymers.

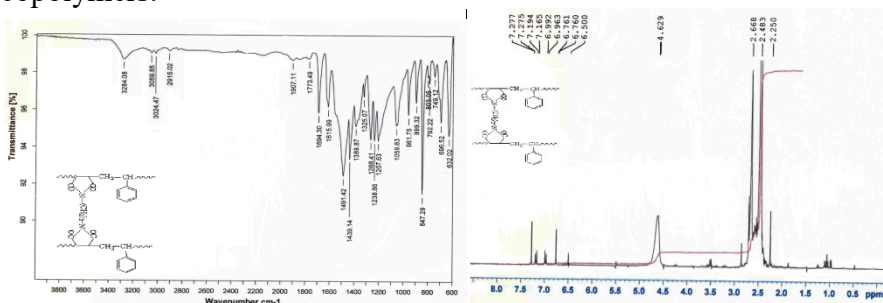


Fig 7. IR and ^1H NMR spectrum of N,N'-(1,2-ethane) bismaleinimide–styrene copolymers

Microstructural parameters of the copolymers were calculated based on the values of copolymerization constants (Table 2). Relative activity values of monomers are $r_1 = 0.02$; $r_2 = 18.2$ for ethylene-bismaleimide – styrene pair and $r_1=0.01$; $r_2 = 12.5$ for phenylene bismaleimide-styrene pair. As is evident from Table 2, relative activity of St monomer (M_2) is higher in copolymerization of

these monomers. Therefore, composition of copolymers is rich in St units.

As the amount of BMI monomers in the mixture of monomers increases, the reaction rate and polymerization degree decrease. This is due to the fact that St possesses a high activity, on the other hand, a high rate of polymerization, while BMI is passive in radical polymerization, but actively participates in the reaction of chain transfer through the monomer.

The studies revealed that adding MI units to polystyrene macrochain has higher antimicrobial effect on the cultures of selected microorganisms in the concentrate, but a lower antimicrobial effect in sterile distilled water-soluble form. In comparison, effect of all the studied substances on the fungal cells (*C. albi-cans*) was more effective.

The antibacterial properties of Styrene–EBMI and Styrene–FBMI copolymers have been studied and it has been determined that the obtained polymeric materials have antibacterial and antifungal properties.

7. Research of applications of synthesized compounds

The possibility of using mono- and bisimides of cyclic dicarboxylic acids as modifiers, modifiers-plasticizers, antipyrenes for ED-20 resin has been revealed. The purchased adhesive-quality composition can be used in the manufacture of photographic, optical and semiconductor devices in the defense industry. HCDCUDA bisimide (43) to obtain thermo - and refractory polymer adhesive composition; N,N'-(3,3'-dimethoxy-4,4'-diphenylmethane) bisimides of 1,4,5,6,7,7-hexachlorobicyclo[2.2.1]hept-5-ene-2,3- dicarboxylic acid- (27) and 1,2,3,4,11,11-hexachlorotricyclo[6.2.1.0^{5,10}]undes-2-ene-7,8-dicarboxylic acid (40) antipyrene and N, N'-(1,6-hexane) dianhydride of endo, endo, endo-1,2,3,4-tetrachlorotricyclo-[6.2.2.0^{5,10}]dodes-2-ene-7,8-dicarboxybisimide-11,11',12,12'-tetra-carboxylic acid (72) can be used as a modifier.

CONCLUSIONS

1. One-stage effective synthesis methods were developed for mono- and bisimidodienes of polychlorinated cyclic dicarboxylic acids on the basis of 1,2,3,4,7,7-hexachlorobicyclo[2.2.1]hept-2-en-5,6-dicarboxylic acid (HCBCHDA) and 1,2,3,4,11,11-hexachlorotricyclo[6.2.1.0^{5,10}]-undec-2-ene-7,8-dicarboxylic acid (HCTCUDA) bisimides diene adducts in the presence of isonicotinic acid. Existence of a diene system in the obtained mono- and bisimidodienes was identified by UV, NMR, IR spectroscopy and methods of X-ray structural analysis [3, 5, 7, 14, 16, 18, 19, 21, 28, 39].
2. Diene condensation of maleic, cis-4-THDA anhydride and bisimides with TCCHDDA and TCBCDDDA anhydridodienes and N, N'-alkyl-(aryl)bisimides were studied. It was determined that the reaction occurs according to Alder rule and results in obtaining dianhydrides and bisimidodianhydrides of chlorinated tricyclic acids by stereospecific *endo, endo, endo*-combination of rings. Polychlorinated cyclic polyimides based on bisimidodienes and mixed diene-dienophiles were obtained [1, 2, 6, 9, 10, 12, 13, 15, 17, 28].
3. For the first time, mixed bisimides with both diene and dienophile fragments in one molecule were obtained from HCBCHDA and HCTCUDA aminoimides, and the structure of bisimides was confirmed by IR and NMR spectroscopy [20, 22-25].
4. Effect of the composition and structure of cyclic bisimides obtained via Diels-Alder reaction on the antibacterial properties was studied. It was shown that the synthesized *endo, exo*-1,2,3,4,11,11-hexachlorotricyclo[6.2.1.0^{5,10}]undec-2-en-7,8-dicarboxylic acid N-[2-maleinimido]ethylbisimide and 1,4,5,6,7,7-hexachlorobicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid N-[4-maleinimidobenzyl]phenylbisimide possess higher antimicrobial and fungicidal properties [29].
5. Polychlorinated cyclic polyimides based on bisimidodienes and mixed diene-dienophiles were synthesized. Polycondensation was carried out based on TCCHDDA and TCBCDDDA N, N'-

(1,2-ethane)bisimidodienes, N, N'-(diphenylmethane) bismaleinimides and mixed diene-dienophiles by Diels-Alder reaction, known spatial structured, moving-ladder polyimides were obtained. The results of DTA and TGA methods prove their resistance to 390-400°C [9, 15, 21, 26, 27].

6. Effect of monomer concentration, reaction time and reaction temperature on the viscosity of the obtained polyimides was studied and optimal conditions were determined for the synthesis of polyimides. It was determined that polyimides possess a higher viscosity when the reaction is carried out at 150°C for 6 h and the concentration of monomers is 20 wt% for bisimidodienes and bismaleinimides, but 25 wt% for diene-dienophiles [15, 21, 27].
7. For the purpose of obtaining cyclic bisimide group-containing polymers, copolymerization reactions of (bis)maleimides with allyl ether of salicylic acid and styrene were carried out and biological activity of the obtained copolymers were studied. The results of the studies reveal that copolymerization reaction of (bis) maleimides with St results in obtaining blockcopolymers, but copolymerization reaction with allyl ester of salicylic acid results in obtaining sequence-structure copolymers [36, 37].
8. New composite materials with PE, PP, ABS and “Noryl” matrix were obtained by adding newly synthesized bisimides into the developed composite mixtures in extrusion stage. It was determined that adding 0.1-1.0 wt% of bisimides to PE-, PP-, ABS- and Noryl based-CM in extrusion stage doesn't affect or slightly affect their physical and mechanical properties. The obtained CM possesses antibacterial properties, and the modified CM has fungicidal properties [29-35].
9. Synthesized compounds and the composite materials may be applied in production of long-acting antibacterial polymer materials used in agriculture and medicine; as modifiers in production of adhesive compositions for optical devices; semi-volcanic retarders-; adhesion additives to adhesive composition for photoreceptors; antipyrene for obtaining refractory materials

from medium-pressure polyethylene composition and ED-20 resin [8, 11, 29-35].

The following papers have been published on the dissertation materials:

1. Alikhanova, A.I., Salakhov, M.S., Umaeva, V.S., Allahverdiev, M.A. Synthesis and stereochemistry of imides based on cyclic dicarboxylic acids and urea derivatives // Scientific conference dedicated to the 90th anniversary of corresponding member of ANAS Z.Sh. Zulfugarov, – Baku, – 2004, – p. 184.
2. Alikhanova, A.I. Stereospecific synthesis of anhydride-imides, imido-anhydrides and diimides of tricyclic tetracarboxylic acids // Materials of the scientific conference of graduate students of ANAS, – Baku, – 2005, – p. 82.
3. Salakhov, M.S., Umaeva V.S., Alikhanova, A.I. Chlorinated N,N'-bisimidodienes of cyclic dicarboxylic acids - as monomers for nonflammable polyimides // VI Baku International Y.Mamedaliyev petrochemistry conference, – Baku, – 2005, – p. 141.
4. Salakhov, M.S., Umaeva V.S., Alikhanova, A.I. Synthesis of polychlorinated imido- and bis-imidodienes of dicarboxylic acids // III International Scientific Conference on "Fine Organic Synthesis and Catalysis" dedicated to the 85th anniversary of the ASOA, – Baku, – 2005, – p. 62-63.
5. Alikhanova, A.I. Synthesis of diimides of 1,2,3,4-tetrachlorotricyclo[6.2.2.0^{5,10}]dodes-2-en-7,8,11,12-tetracarboxylic acids // Materials of the scientific conference of graduate students of ANAS, – Baku, – 2006, – p. 219.
6. Salakhov, M.S. Synthesis of N-substituted imides 1,2,3,4-tetrachlorocyclic[4.4.0]deca-2,4-diene-8,9-dicarboxylic acids / M.S. Salakhov, V.S. Umaeva, A.I. Alikhanova, M.M.Ibragimova // Azerbaijan Chemical Journal, – 2006, – № 4, – p. 86-88.
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- tetracarboxylic acids // II Republican scientific conference "Young chemist", – Baku, – 2006, – p. 38.
8. Salakhov, M.S. New cryogenic epoxy adhesive composites for photodetectors / M.S.Salakhov, V.S.Umaeva, A.İ.Alikhanova // International Polymer Science and Technology, – 2007. – Vol. 34, – №5, – p. 1-3.
 9. Alikhanova, A.I. Synthesis of stereospecific hinged ladder polyimides // Materials of the scientific conference of graduate students of ANAS, – Baku, – 2007, – p. 135-138.
 10. Salakhov, M.S., Umaeva V.S., Alikhanova, A.I. Synthesis methods for tetrachloride-cyclohexadiene-fragmented imides of dicarboxylic acids // Scientific conference dedicated to the 100th anniversary of Academician H. Afandiyev, – Baku. – 2007, – p. 268-269.
 11. Salakhov, M.S. Fire-resistant epoxy compositions / M.S.Salakhov, V.S.Umaeva, A.I.Alikhanova // J. "Plast. mass", – 2008, – №7, – p.12-13.
 12. Salakhov, M.S., Umaeva V.S., Alikhanova, A.I. Synthesis of polychlorinated bisimides 1,2,3,4,11,11-hexachlorotricyclo [6.2.1.0^{5,10}]undec-2-en-7,8-dicarboxylic acids // Scientific conference dedicated to the 100th anniversary of Academician M.F. Nagiyev, – Baku, – 2008, – p. 143.
 13. Salakhov, M.S. Synthesis of polychlorinated unsaturated cyclic dicarboxylic acid imides / M.S.Salakhov, V.S.Umaeva, A.I.Alikhanova // Russian J. Org. Chemistry, Sankt-Peterburg, – 2008, Vol.44, № 10, – p. 1438-1443.
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 15. Salakhov, M.S. Synthesis of stereospecific hinged ladder polyimides by Diels-Alder reaction / M.S.Salakhov, V.S.Umaeva, A.I.Alikhanova // J. of Appl. Chem., – 2009, – Vol. 82, – Issue 2, – p. 323-327.

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17. Salakhov, M.S., Umaeva, V.S., Alikhanova, A.I. Synthesis and stereochemistry of N,N'-Oxy(methylene)-diphenylene-bridged polychlorinated cage-like tetracarboxylic acid imides // Russian J. Org. Chemistry, Sankt-Peterburg, – 2010. – Vol. 46, – № 10, – p. 1501-1505.
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