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Antibacterial Polyurethane Adhesives for Medical Applications

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Abstract

In this study, polyurethane prepolymers and two-component adhesive systems with antibacterial properties were synthesised and characterised for potential biomedical applications as tissue adhesives. The developed materials were designed to operate in moist biological environments and to provide simultaneous adhesive and antibacterial functions. The viscosity of the obtained prepolymers, measured using a Brookfield viscometer, ranged from 2.30 to 3.36 Pa·s, indicating favourable rheological properties for dosing, mixing, and application on tissue surfaces. The chemical structure of the crosslinked adhesives was confirmed by FTIR spectroscopy, which revealed characteristic urethane bands and the absence of isocyanate groups, indicating complete conversion during curing. Contact angle measurements demonstrated the hydrophilic nature of the materials, suggesting good potential for adhesion to wet biological tissues. Mechanical characterisation showed medium hardness (medium-soft materials) and high adhesive strength exceeding 5 MPa, confirming suitable mechanical performance for tissue bonding applications. Antibacterial testing indicated that the incorporation of a biopolymer-based antimicrobial agent resulted in strong biocidal activity, outperforming conventional silver-based additives. The results demonstrate that the developed polyurethane-based adhesive systems combine favourable mechanical, physicochemical, and antibacterial properties, making them promising candidates for surgical applications.

Keywords:

antimicrobial activity, bio-based antimicrobial agent, montmorillonite, polylysine

1. INTRODUCTION

The development of modern biomaterials used in surgery in recent years has led to a significant increase in interest in tissue adhesives as an alternative to conventional wound closure methods, such as surgical sutures and mechanical staplers. Particular attention has been paid to polyurethane adhesives, which, due to their high biocompatibility and the possibility of precise tailoring of mechanical properties, are finding increasingly widespread applications in medicine. Owing to the wide range of available raw materials, it is possible to obtain materials with suitable flexibility, adhesion to moist tissues, and controlled biodegradation, making them a promising group of biomaterials intended for surgical applications. Contemporary research is focused not only on improving the mechanical properties of these materials but, above all, on endowing them with specific functionalities, including antimicrobial activity and the ability to support tissue regeneration processes [1–4].

One of the major challenges in modern surgery remains wound infections, which significantly increase the risk of postoperative complications, prolong hospitalisation time, and raise treatment costs. This issue is particularly important in procedures performed within mucosal membranes and tissues with a high degree of bacterial colonisation, such as the oral cavity, nasopharynx, and upper respiratory tract. Consequently, there is growing interest in adhesive materials that simultaneously exhibit haemostatic, sealing, and antibacterial properties [5, 6].

Tissue adhesives are currently used in numerous surgical fields, including cardiac surgery, neurosurgery, general surgery, otorhinolaryngology, and dentistry. In head and neck surgery, adhesive materials are employed to support haemostasis, seal tissues, and reduce postoperative complications following procedures involving mucosal membranes. They are of particular importance in procedures such as adenotomy, tonsillectomy, endoscopic surgery of the nose and paranasal sinuses, and reconstruction of mucosal defects, where effective protection of the surgical site may contribute to reducing the risk of secondary bleeding, limiting local inflammation, and improving the healing process. The application of modern bioadhesive materials may also contribute to reducing postoperative pain and shortening patient recovery time [7].

An important direction in the development of modern tissue adhesives is the design of materials capable of functioning effectively in moist environments, which are characteristic of most tissues in the human body. Adhesive systems based on cyanoacrylates, albumin-based adhesives, or fibrin glues, despite their widespread clinical use, exhibit several limitations, such as insufficient flexibility or mechanical strength, the risk of local cytotoxicity, and the lack of antibacterial activity. Polyurethane adhesives represent an alternative class of materials characterised by high adhesion, suitable mechanical strength, and a controlled degradation rate [8].

In recent years, the concept of 'smart' polyurethane biomaterials has also been intensively developed. These materials are designed not only to provide mechanical wound closure but also to actively support tissue regeneration through the controlled release of bioactive substances, anti-inflammatory effects, and modulation of the body's immune response. The development of nanomaterial technologies and advanced polyurethane surface functionalisation methods opens new possibilities for designing bioactive surgical adhesives with multifunctional therapeutic effects. For this reason, antibacterial polyurethane adhesives are currently considered one of the most promising research directions in the field of modern biomedical materials and may play an important role in the future development of, among others, minimally invasive surgery and regenerative medicine [9].

In the case of polyurethane adhesives, these properties can be achieved through modification with quaternary ammonium salts, metal nanoparticles, or compounds containing hydrophilic groups. Such approaches make it possible to reduce the adhesion and proliferation of microorganisms on the biomaterial surface and decrease the risk of bacterial biofilm formation [10, 11].

Metal nanoparticle additives currently rely mostly on silver, nanosilver, or silver compounds. Some silver-free systems using nano zinc oxide, or nano titanium dioxide have been proposed. However, metal-based nanoparticles may cause aggregation, phase separation, and toxicological issues [12]. Also, combinations of silver with organic antimicrobial agents, such as quaternary ammonium compounds (QACs), are employed. QACs have the chemical structure $R_1R_2R_3R_4\ N^+X^-$ with at least one long hydrophobic hydrocarbon chain substituent that can penetrate into microbes to disrupt cell membranes. The ammonium group of QACs attracts negatively charged microbes [13]. QACs are an example of biocides active against both bacteria and enveloped viruses [14]. The interference of the lipophilic hydrocarbon chain with the envelope may potentially reduce virus infectivity [15]. A notable drawback of simple QACs, particularly those widely used, is that bacteria may develop resistance to these compounds [16].

Due to the development of bacterial resistance to these compounds, alternative solutions are being sought. In the following work, we employed the biopolymer polylysine (PLY) intercalated on montmorillonite (MM) as an antibacterial agent. Therefore, the aim of this work was to develop and characterise a polyurethane adhesive with a bio-based antibacterial agent. The study focused on understanding how the polyurethane structure and bio-based antimicrobial agent affect the mechanical and antibacterial properties of the adhesives.

2. MATERIAL AND METHODS

2.1. Materials

The following materials were used in this study:

- isophorone diisocyanate (IPDI), Evonic-Degussa (Germany),
- polyoxyethylene glycol M_n = 300 g/mol (PEG300), PCC Rokita (Poland),
- polyoxypropylenediol M_n = 750 g/mol (Rokopol 750D), PCC Rokita (Poland),
- polyoxypropylenediol M_n = 2000 g/mol (Rokopol D2002) PCC Rokita (Poland),
- triethanolamine (TEA), Aldrich (Switzerland),
- glycerine (GLY), Aldrich (Switzerland),
- dibutyltin dilaurate, Aldrich (Switzerland),
- tin octoate, Aldrich (Switzerland),
- silver phosphate (Ultra Fresh CA-16), Lexington (Malaysia),
- ε-polylysine (PLY) >95%, Mark Nature (Fullerton, CA, USA),
- sodium montmorillonite nanoclay (MM) (Sigma-Aldrich).

The intercalation of polylysine on montmorillonite was performed at the Łukasiewicz Research Network – Institute of Industrial Chemistry in Warsaw. Polyoxyethylene glycol, polyoxypropylenediols, triethanolamine, and glycerine, were dried at 110°C for 3 hours and stored over 4A molecular sieves. The other reagents were used as received.

Prepolymer synthesis

IPDI was placed in a three-necked flask equipped with a heating mantle, reflux condenser, dropping funnel, thermometer, mechanical stirrer, and nitrogen inlet tube and heated to 40°C. Polyol was then slowly added dropwise over 30 min. After adding the polyol, the reaction was carried out at 75°C until the –NCO group content reached the theoretically calculated value (about 2 hours). The reaction progress was monitored by determining the free –NCO group content using the acidimetric method. The obtained prepolymer was cooled to room temperature and poured into a container with a tight closure. The qualitative and quantitative composition of polyurethane prepolymers are presented in Table 1.

Table 1

Qualitative and quantitative composition of polyurethane prepolymers

Prepolymer symbol	Isocyanate	Isocyanate content [g/mol]	Polyol	Polyol content [g/mol]	–NCO content [%]
IPDI/PEG300	IPDI	22.2/0.1	PEG 300	15.0/0.05	11.2
IPDI/750D	IPDI	22.2/0.1	Rokopol 750D	37.5/0.05	7.2
IPDI/PEG300/D2002	IPDI	22.2/0.1	PEG 300 Rokopol D2002	7.5/0.025 50.0/0.025	5.3

Preparation of adhesive samples

Samples of adhesives were obtained by mixing the prepolymers first with the antimicrobial agent (silver phosphate or PLY/MM) and then with a cross-linking agent and the catalyst. The cross-linking agents were glycerine or triethanolamine. The amount of cross-linking agent was adjusted to obtain a molar ratio of –NCO:OH groups of 1.10:1. Dibutyltin dilaurate or tin octoate was used as the catalyst at a concentration of 0.02%. All ingredients were mixed with a mechanical stirrer in a closed flask at room temperature for 2 minutes. The mixture was then poured into silicone moulds and allowed to cure in a desiccator with 90% humidity. Gelation began after approximately 10 minutes, and curing occurred after approximately 30 minutes, regardless of the composition.

2.2. Methods

The adhesive was characterised using the following methods.

Gel permeation chromatography (GPC)

Gel permeation chromatography (GPC) studies were performed using an Agilent 1100 Series liquid chromatograph equipped with a G1379A degasser, a G1311A four-channel QuatPomp pump, a G11316A column thermostat, a VARIAN PLGEL 5#M MIXED-D chromatography column (*dp* = 5 µm, column dimensions 300 × 7.5 mm) and dedicated precolumn, an RID G1362A refractometric detector, and a valve injector with a 20 µL loop. Analyses were performed at 35°C with a mobile phase (THF) volumetric flow rate of 1 ml/min.

Viscosity tests

A Brookfield CAP 2000+ viscometer was used to test viscosity.

Hardness tests

Hardness tests was carried out in accordance with the PN-EN ISO 868 standard, using a Shore A hardness tester.

Strength tests

The tensile strength and percentage elongation of the hardened castings were determined on an INSTRON 5967 universal testing machine. The measurement was performed under a load of 5 kN and a crosshead speed of 20 mm/min at room temperature.

Water contact angle tests

Water contact angle measurements were performed using a Data Physic optical goniometer, model OCA 15, according to the ISO 19403 standard.

Fourier-transform infrared spectroscopy (FTIR)

FTIR analysis was performed using a Nicolet™ iS™ 10 FTIR spectrometer using the ATR technique.

Antibacterial tests

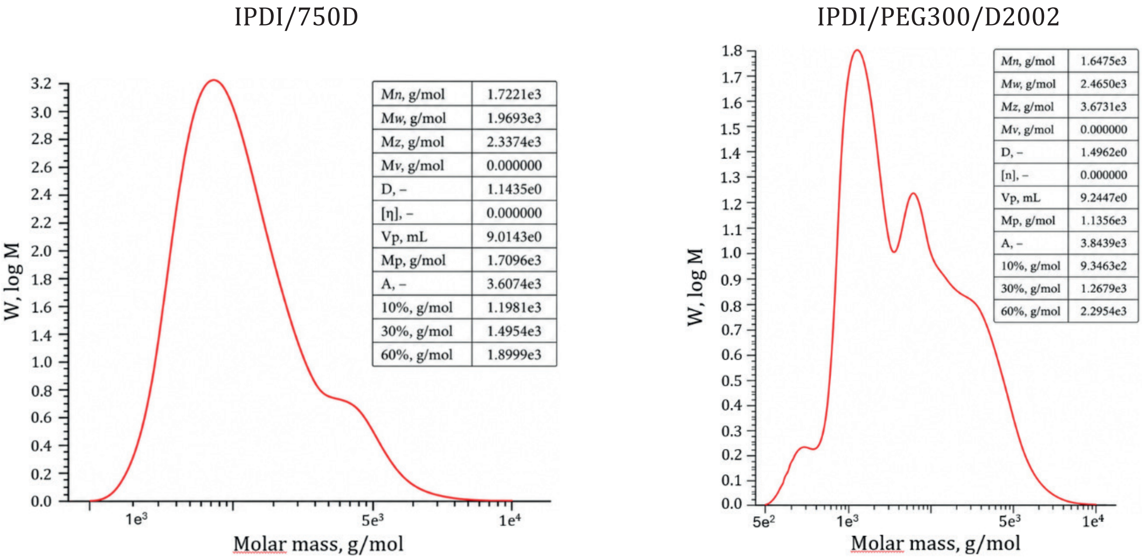
Antibacterial properties were tested according to the ISO 22196 standard on E. Coli and S. Aureus bacteria.

Adhesion tests

The adhesion of the adhesive to pig tissue was tested by the pull-off method using a PosiTest AT-A Automatic Adhesion Tester.

3. RESULTS AND DISCUSSION

Isophorone diisocyanate (IPDI) was used for the synthesis of the adhesives due to its cycloaliphatic structure, which imparts flexibility and exhibits lower toxicity compared to aromatic polyisocyanates. Aliphatic polyoxy glycols with different molecular weights were selected as the polyol component. The molar ratio of isocyanate to polyol was 2:1. During the synthesis of the IPDI/PEG300 prepolymer, uncontrolled gelation of the product occurred. The molecular weight distribution was analysed using gel permeation chromatography (GPC). Examples of GPC chromatograms are shown in Figure 1.


IPDI/750D

IPDI/PEG300/D2002

M*n*, g/mol	1.7221e3
M*w*, g/mol	1.9693e3
Mz, g/mol	2.3374e3
M*v*, g/mol	0.000000
D, –	1.1435e0
[η], –	0.000000
Vp, mL	9.0143e0
Mp, g/mol	1.7096e3
A, –	3.6074e3
10%, g/mol	1.1981e3
30%, g/mol	1.4954e3
60%, g/mol	1.8999e3

M*n*, g/mol	1.6475e3
M*w*, g/mol	2.4650e3
M*z*, g/mol	3.6731e3
M*v*, g/mol	0.000000
D, –	1.4962e0
[n], –	0.000000
Vp, mL	9.2447e0
Mp, g/mol	1.1356e3
A, –	3.8439e3
10%, g/mol	9.3463e2
30%, g/mol	1.2679e3
60%, g/mol	2.2954e3

Fig. 1. GPC chromatograms of IPDI/750D and IPDI/PEG300/D2002 prepolymers

Based on the obtained GPC chromatograms, the average molecular weights and dispersity index of the prepolymers were determined (Table 2).

In the chromatograms, in addition to the signal corresponding to the main product resulting from the reaction stoichiometry (a prepolymer with the IPDI–polyol–IPDI structure), products with higher molecular weights were also observed, which is typical for prepolymer synthesis via polyaddition reactions. The dispersity index (PD) values were 1.14 and 1.5, indicating a relatively narrow molecular weight distribution. Both obtained prepolymers exhibited relatively low viscosity values (Table 2), which is advantageous during dosing, mixing, and spreading of the adhesive on tissues.

Glycerine or triethanolamine, together with organotin catalysts, was used as the crosslinking agent for the adhesives. The molar ratio of prepolymer –NCO groups to the OH groups of the crosslinking agent was 1.10:1. Attempts to crosslink the adhesives in the presence of amine catalysts resulted in product foaming due to accelerated catalysis of the competing reaction with moisture, leading to carbon dioxide evolution. The course of the adhesive crosslinking process using glycerine is shown in Figure 2.

The crosslinked IPDI/PEG300/D2002 sample was brittle and prone to fracture; therefore, only the IPDI/750D/GLY and IPDI/750D/TEA compositions were selected for further studies. Formulations containing silver phosphate and polylysine intercalated in montmorillonite (PLY/MM) were prepared (Table 3).

After crosslinking, the best properties were exhibited by the glycerine-crosslinked formulations prepared in the presence of dibutyltin dilaurate as the catalyst, namely IPDI/750D/GLY/Ag/DSn and IPDI/750D/GLY/PLY/MM/DSn (Fig. 3).

Table 2
GPC results and viscosity of polyurethane prepolymers

Prepolymer	M_n [g/mol]	M_w [g/mol]	PD	Viscosity [Pa·s]
IPDI/750D	1722.1	1969.3	1.14	2.30
IPDI/PEG300/D2002	1647.5	2465.0	1.50	3.36

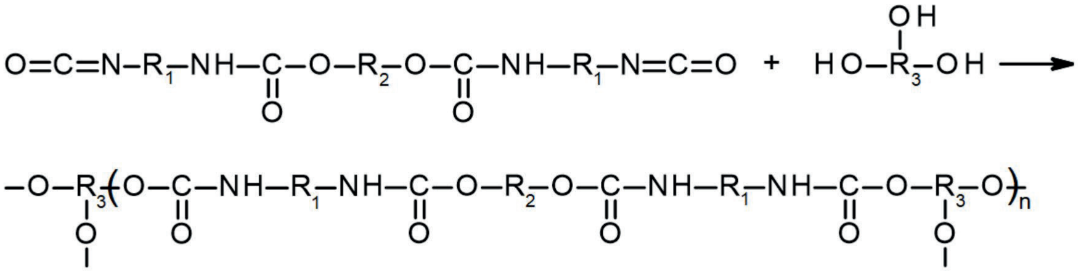


Fig. 2. Course of the prepolymer crosslinking reaction using glycerine or triethanolamine

Table 3
Qualitative and quantitative composition of polyurethane adhesives

No.	Sample symbol	Prepolymer	Curing agent	Antimicrobial agent [%]	Catalyst [%]
1	IPDI/750D/GLY/Ag/OSn	IPDI/750D	glycerine (GLY)	Ag_3PO_4 (Ag), 0.5	tin octoate (OSn)/0.02
2	IPDI/750D/GLY/PLY/MM/OSn	IPDI/750D	glycerine (GLY)	PLY/MM, 2.0	tin octoate (OSn)/0.02
3	IPDI/750D/TEA/Ag/DSn	IPDI/750D	triethanolamine (TEA)	Ag_3PO_4 (Ag), 0.5	dibutyltin dilaurate (DSn)/0.02
4	IPDI/750D/TEA/PLY/MM/DSn	IPDI/750D	triethanolamine (TEA)	PLY/MM, 2.0	dibutyltin dilaurate (DSn) 0.02
5	IPDI/750D/GLY/Ag/DSn	IPDI/750D	glycerine (GLY)	Ag_3PO_4(Ag), 0.5	dibutyltin dilaurate (DSn) 0.02
6	IPDI/750D/GLY/PLY/MM/DSn	IPDI/750D	glycerine (GLY)	PLY/MM, 2.0	dibutyltin dilaurate (DSn) 0.02

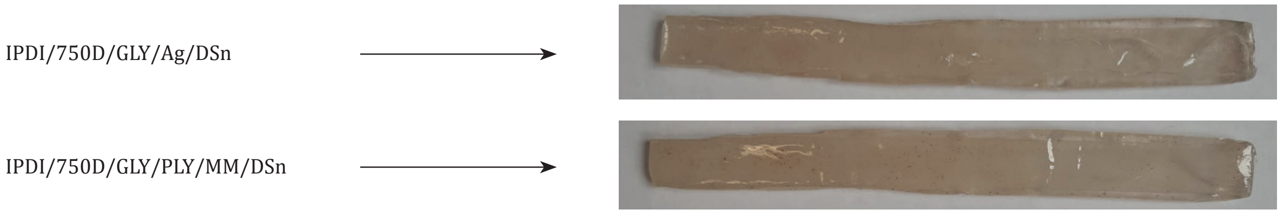


Fig. 3. Appearance of adhesive samples after cross-linking

The structure of the crosslinked adhesives was confirmed using FTIR spectroscopy (Fig. 4).

The band at 3300 cm^{-1} is characteristic of N–H stretching vibrations, the band at 1700 cm^{-1} corresponds to C=O stretching vibrations, while the signal in the 1550 cm^{-1} region is attributed to N–H bending vibrations, confirming the presence of urethane groups. No absorption band at 2200 cm^{-1}, assigned to isocyanate groups, was observed in the spectra, indicating their complete conversion into urethane linkages.

The measured Shore A hardness and tensile strength values of the adhesives are presented in Figure 5 and Table 4.

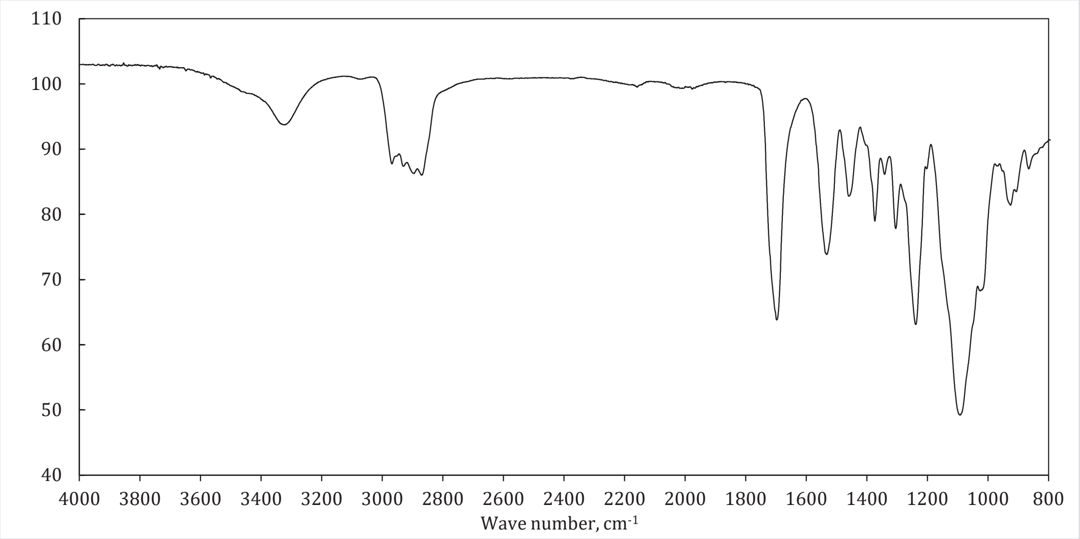


Fig. 4. FTIR spectrum of IPDI/750D/GLY/Ag/DSn adhesive

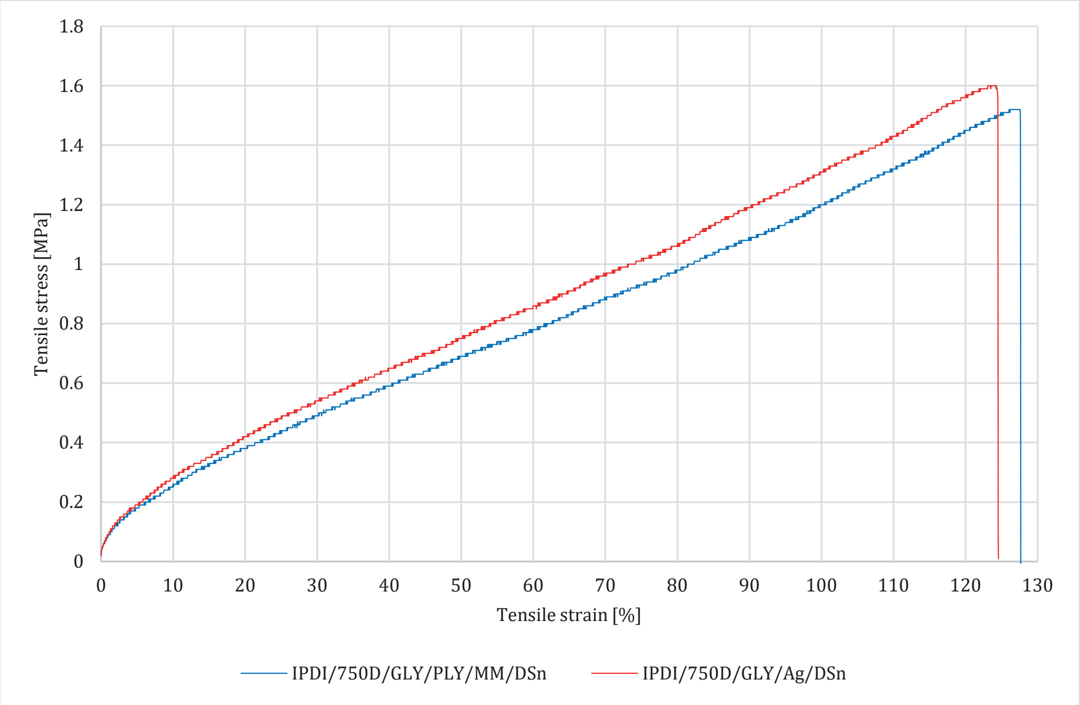


Fig. 5. Tensile stress–strain curves of adhesives IPDI/750D/GLY/Ag/DSn and IPDI/750D/GLY/PLY/MM/DSn

Table 4
Summary of Shore A hardness and tensile strength values of adhesives

Sample symbol	Hardness Shore A	Tensile strength [MPa]	Strain at break [%]	Young's Modulus [GPa]
IPDI/750D/GLY/Ag/DSn	35	1.60	124	1.96
IPDI/750D/GLY/PLY/MM/DSn	36	1.52	128	1.95

The hardness and tensile strength results indicate good mechanical strength and elasticity of the adhesives, which is highly advantageous for accommodating tissue movement.

The tensile bond strength of porcine tissue joints was 5.94 MPa (Fig. 6). At this value, failure occurred first at the fixture (grip detachment), while neither adhesive nor cohesive failure of the tissue specimens was observed. This indicates that the synthesised adhesive is resistant to tensile stresses exceeding the measured value.

Fig. 6. A sample of bonded porcine tissues prepared for pull-off adhesion testing

The contact angle values of all four adhesives were below 90°, indicating that the obtained materials are hydrophilic (Fig. 7). This property may promote improved adhesion of the adhesive to skin. Comparing the contact angle values, it can be observed that the lowest value was obtained for the glycerine-cured adhesive containing silver phosphate as the antibacterial additive. This is attributed to the more hydrophilic nature of both glycerine and Ag_3PO_4 compared to triethanolamine and polylysine.

The results of the antibacterial tests are presented in Table 5.

The conducted studies indicate that polylysine exhibits superior antibacterial properties. The percentage reduction values for this agent exceeded 99%, indicating that *Escherichia coli* and *Staphylococcus aureus* cells were almost completely eliminated. The percentage reduction value for Ag_3PO_4 reached 38.55%, which indicates approximately a threefold lower effectiveness of this commonly used biocide. Higher antibacterial activity of polylysine compared to Ag_3PO_4 was also observed in the case of modifications of other polymers [17].

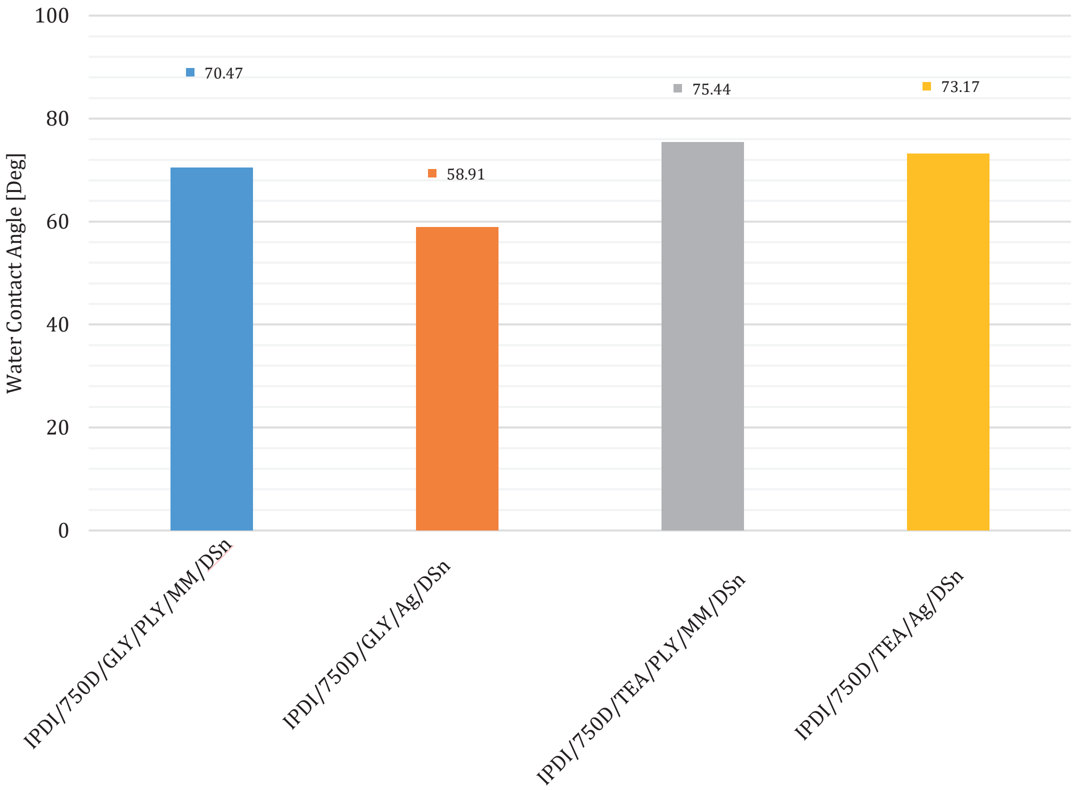


Fig. 7. Water contact angle of IPDI/750D prepolymer-based adhesive

Table 5
Summary of antibacterial activity results

Sample symbol	Antimicrobial agent	Bacteria	% reduction	Log reduction
IPDI/750D/GLY/Ag/DSn	Ag_3PO_4	*S. Aureus*	38.5511	0.21
IPDI/750D/GLY/PLY/MM/DSn	PLY/MM	*E. Coli*	99.9984	4.80
IPDI/750D/GLY/PLY/MM/DSn	PLY/MM	*S. Aureus*	99.7330	2.57

4. CONCLUSIONS

As part of the conducted studies, polyurethane prepolymers and two-component adhesive systems exhibiting antibacterial properties were synthesised. The obtained materials were characterised by favourable rheological properties: the viscosity of the prepolymers, measured using a Brookfield viscometer, ranged from 2.30 to 3.36 Pa·s, indicating good processability, ease of dosing, and applicability to tissue surfaces.

Confirmation of the chemical structure of the crosslinked adhesives was obtained through FTIR spectroscopy analysis, which demonstrated the presence of characteristic urethane groups and the complete conversion of isocyanate groups. Contact angle measurements revealed the hydrophilic nature of the obtained materials, which may promote good adhesion to moist biological tissues.

Mechanical testing showed that the developed adhesives exhibit medium hardness (so-called medium-soft materials), which is a desirable feature in medical applications where mechanical compatibility with tissues is essential. At the same time, the adhesive bond strength exceeding 5 MPa indicates high adhesion to tissues and effective sealing of the application site.

The conducted tests also demonstrated that the applied biopolymer-based antibacterial agent exhibits very high biocidal efficiency, representing a promising alternative to commonly used silver compounds.

Future research will focus on optimising the adhesive composition in order to tailor its mechanical properties to different types of tissues. It will also be important to extend the study to investigate biodegradation under in vitro and in vivo conditions. Additionally, determining the release kinetics and duration of antibacterial activity of the additives in a biological environment will be a key aspect of further investigations.

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