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AN ANALYSIS OF SELECTED FUNCTIONAL CHARACTERISTICS OF TEMPORARY RESTORATIVE MATERIALS USED IN CONSERVATIVE DENTISTRY

ANALIZA WYBRANYCH CECH UŻYTKOWYCH TYMCZASOWYCH MATERIAŁÓW ODTWÓRCZYCH STOSOWANYCH W STOMATOLOGII ZACHOWAWCZEJ

Key words: biomaterials, temporary dental filling materials, changes of surface structure, mass loss.

Abstract

An important factor in endodontic therapy is forming a tooth seal as a temporary restoration, which prevents leakage of medicines into the oral cavity, infiltration of microorganisms, and contamination of the root canal system by fluids or food debris. Suitable physicochemical analysis and usability evaluation of restorative materials, i.e. microhardness, abrasion and compression resistance, surface structure, behaviour at different temperatures, the sealing of tooth margins, the ease of insertion and removal, allow one to choose the material that will be most favourable for medical treatment. The aim of the study is to determine, in vitro, changes of the surface structure that arise over time in commonly used preparations for temporary dental fillings. The results showed significant structural changes occurring over time, especially at a temperature of 37°C (inflammation to be), which, for a long-term use, may have a negative impact on the treatment.

Słowa kluczowe: biomateriały, tymczasowe wypełnienia stomatologiczne, zmiany struktury powierzchni, ubytek masy.

Streszczenie

Istotnym czynnikiem w prowadzonej terapii endodontycznej jest utworzenie uszczelnienia zęba w postaci tymczasowego uzupełnienia, które zapobiega wyciekaniu leków do jamy ustnej, dostawaniu się drobnoustrojów oraz zanieczyszczaniu kanałów korzeniowych przez płyny czy resztki pokarmu. Przeprowadzenie odpowiedniej analizy fizykochemicznej, a także oceny użytkowej materiałów odtwórczych, tj. mikrotwardości, odporności na ściskanie i ścieranie, struktury powierzchni, zachowania w różnych temperaturach, szczelności brzeżnej, łatwości wprowadzania i usuwania, pozwala na dobór materiału, który będzie najkorzystniejszy dla prowadzonego leczenia. Celem badań było określenie zmian struktury powierzchni powstających w czasie pod wpływem oddziaływania środowiska oraz odporności na zużywania ścierne dla stosowanych preparatów do tymczasowych wypełnień stomatologicznych. Wyniki wykazały istotne zmiany struktury powstające w czasie, szczególnie w temperaturze 37°C (teoretycznie początek stanu zapalnego), co przy dłuższym stosowaniu może mieć negatywny wpływ na prowadzone leczenie.

INTRODUCTION

One of the most important functions of dental filling is the reinstatement and maintenance all of anatomical and physiological functions of the humans stomatognathic system (SS), i.e. chewing, and occlusion. Due to the fact that these materials are designed for use in conditions of body fluids and cyclic pressures depending on the nature of the US functioning, they should feature specific mechanical and tribological properties.

Due to biomedical reasons, the following tasks are required for filling materials:

- Restore the function and reconstruct shape of the tooth;

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- Dental pulp isolation and protection of hard tooth tissues in front of bacterial infections, chemiotoxic, thermal, and osmotic trauma; and,
- Preventing the renewal of carious processes [**L. 2, 5–7, 11**].

Dental fillings may appear as restorative or temporary. The role of restorative materials is to fulfil their function in time duration. Temporary restorations are used temporarily in the period between a tooth preparation and final material insertion. They fulfil the role of protective material for dentin and dental pulp before exterior surrounding [**L. 2, 5–7, 11**].

Mechanical tests are mainly carried out with restorative materials; however, the tests of temporary materials are limited. These materials, like restorative materials, should also have appropriate mechanical properties, including the expected resistance to tribological wear. Due to biomedical reasons, temporary filling materials ought to be characterized by indifference to applied drugs or local tissues, high ease of pasting, putting on, and removing from cavity, fast hardening, high marginal integrity, and the lack of thermal conductivity [**L. 1, 3, 5, 12–14**].

THE ASSESSMENT OF CHANGES IN FUNCTIONAL CHARACTERISTICS OF DENTAL RESTORATIVE MATERIALS – EXPERIMENTAL STUDIES

The aim and research methodology

The aim of the tests was to assess the changes of the surface structure within time influenced by the impacts of environment. The tests were carried out on selected materials for temporary dental fillings in two phases. The first one involved the qualitative assessment of changes and the control of mass loss within time influenced by the impacts of environment. The specimens were placed in artificial saliva and in a solution imitating inflammation at temperatures of 36.6°C and 37°C without free access to oxygen. The second phase consisted of the tribological tests and determined the mass loss on defined friction distance.

The first phase of the tests was to carry out at the laboratory conditions on a specimens made of the temporary filling materials commonly used by dentists (i.e. in the treatment of the tooth pulp disease) of which crucial features are tight marginal adhesion, and resistance to changes in the surface structure and abrasion. Simultaneously, after fulfilling their function, it is necessary to remove these materials; therefore, the durability of these materials is comparative [**L. 1, 3, 12, 14**]. On the basis of the composition analysis, practical application (**Table 1**) and a consultation with a dentist, the selection of materials was made.

According to the manufacturer's recommendations, 10 samples were made for each preparation (total 40 specimens) and the average mass was 1g. The specimen's surfaces of each preparation were evaluated with the microscope and then placed into glass containers in one of the solutions of artificial saliva (**Table 2**). They were stored in incubators with constant temperatures. After 7 days, the specimens were dried, weighed, and visually evaluation of the surface condition was made. Then, the same specimens were placed in the solutions and the procedure was repeated after 28 days.

The solutions of artificial saliva were prepared in laboratory conditions based on the PN-EN ISO 10993-15:2009 norm and literature reports

Table 1. The specification of practical application and usage of tested materials
Tabela 1. Opis składu oraz zastosowań badanych preparatów

Material	Specification	Usage
Thymodentin	– the powder with the mixing distilled water content, – the material hardening without saliva access, – zinc-sulphate cement (water dentin), – ingredients: gypsum, zinc sulphate, yellow dextrin, kaolin, zinc oxide, 0,1 % thymol.	– temporary filling material, – cavities filling, – temporary self-dressing (surface losses).
i-Pro	– self-hardening cement ready for immediate use, – the paste hardens while absorbing water from mouth cavity moisture, – ingredients: based on synthetic resins and zinc oxide.	– temporary filling material, – temporary fixation of crowns.
Plastidentin	– a paste product, – the paste binds at the influence of the mouth cavity moisture within an hour, – ingredients: zinc oxide, calcium sulphate, zinc sulphate, polyvinyl acetate and dibutyl phthalate.	– temporary filling material, – for pressure contraindication on the bottom of the cavity.
Zinc oxide eugenol (ZOE)	– pharmaceutical raw material in powder form mixed with eugenol, – the material hardens with the influence of the mouth cavity moisture.	– temporary filling material, – antiseptic effect, – canal sealant, – caries treatment as an intermediate cover.

Table 2. Chemical ingredients of artificial saliva solution [L. 4, 10]
Tabela 2. Skład chemiczny roztworu sztucznej śliny [**L. 4, 10**]

Artificial saliva solution		Artificial saliva solution imitating inflammation	
Compound	Content g/l	Compound	Content g/dm^3
NaCl	0.7	NaCl	0.4
KCl	1.2	KCl	0.4
KH_2PO_4	0.2	Na_2S	0.005
$NaHCO_3$	1.5	$Na_2HPO4 \cdot H_2O$	0.69
Na_2HPO_4	0.26	$CaCl_2 \cdot 2H_2O$	0.795
KSCN	0.33	$(NH_2)2CO_3$	1.0
pH	8.3	$C_3H_6O_3$ 0.1 M	to pH = 2.7

containing researches of dental and orthodontic materials. Testing time was accepted due to the recommended time for usage of these materials by the manufacturers during endodontic treatment, dental consultation and also based on literature data, where the use of zinc-sulphate cement recommended ranges from a few to several days, and ZOE, self-hardening masses, and cements from a few to several weeks is indicated [**L. 2, 4–6, 10**].

The surfaces of the tested specimens were evaluated by the Mitutoyo Quick Scope Vision Measuring Machine with a Qspak ver.10.2 image processing system, with 100x magnification. The mass measurements were made by the use of RADWAG, WAS 220/X analytical balance with an accuracy of ± 0.1 mg.

Tribological tests were carried out on a tribological research stand designed and made at the Faculty of Mechanical Engineering of the University of Science and Technology in Bydgoszcz [**L. 8, 9**], where the tested friction pairs were specimens of temporary filling materials with a 5 mm diameter, and the counter-specimen (plate) was made of 102Cr6 steel. The friction pair worked with a conformal contact and theoretical contact area, approx. 20 mm^2, with the following independent variables:

- Relative speed: 0.5 m/min;
- Frequency of cooperation: 1.5 Hz;
- Load: 10 N; and,
- Environmental conditions of friction: artificial saliva solution.

At a given phase of experimental tests, the controlled magnitude was the loss of mass by the following friction distances: 5 and 10 meters.

Tests results and their discussion

Table 3 summarizes the results of visual evaluation of the tested specimens after 7 and 28 days in the artificial saliva solutions stored at constant temperature of 36.6°C.

Table 3. Observation list results of surface structure changes of tested materials by organoleptic method after 7 and 28 days in artificial saliva solution
Tabela 3. Zestawienie wyników obserwacji zmian struktur powierzchni badanych preparatów metodą organoleptyczną po 7 i 28 dniach w roztworze sztucznej śliny

Materials	Time of tests	Organoleptic evaluation of the material
Thymodentin	7 days	– slight turbidity of the solution, – the odds appeared.
	28 days	– slight turbidity of the solution, – cracks appeared on a four specimens, – the surface of specimens clearly unsteady, – the surface of three specimens became porous.
i-Pro	7 days	– slight turbidity of the solution.
	28 days	– slight turbidity of the solution, – slight odds appeared on the surface.
Plastidentin	7 days	– slight turbidity of the solution, – on the upper surface (no contact with the glass) a clear crack appeared from the centre of the sample.
	28 days	– slight turbidity of the solution, – no visual changes compared to the results obtained after 7 days.
Zinc oxide eugenol (ZOE)	7 days	– slight turbidity of the solution, – the surface of one specimen clearly unsteady.
	28 days	– slight turbidity of the solution, – the surface of specimens clearly unsteady, – at the one of specimens appeared a crack.

In case of each material, a slight turbidity of the solution was observed, which indicates the dissolution of material. Comparison of the specimens' surface conditions after their preparation with the conditions of the surface after the tests showed significant dynamic growth changes of the material structure surface changes within time. Significant odds and cracks appeared in Thymodentin and ZOE materials (**Fig. 1**). These may be the result of the binding method of material components (with and without saliva access) and the effect of material hydration on its stability. On the entire surface of Plastidentin paste ready for direct applications, after 7 days, the clear crack from the centre of the specimen (**Fig. 2**) appeared. Concerning ready for use i-Pro cement, observed changes were insignificant.

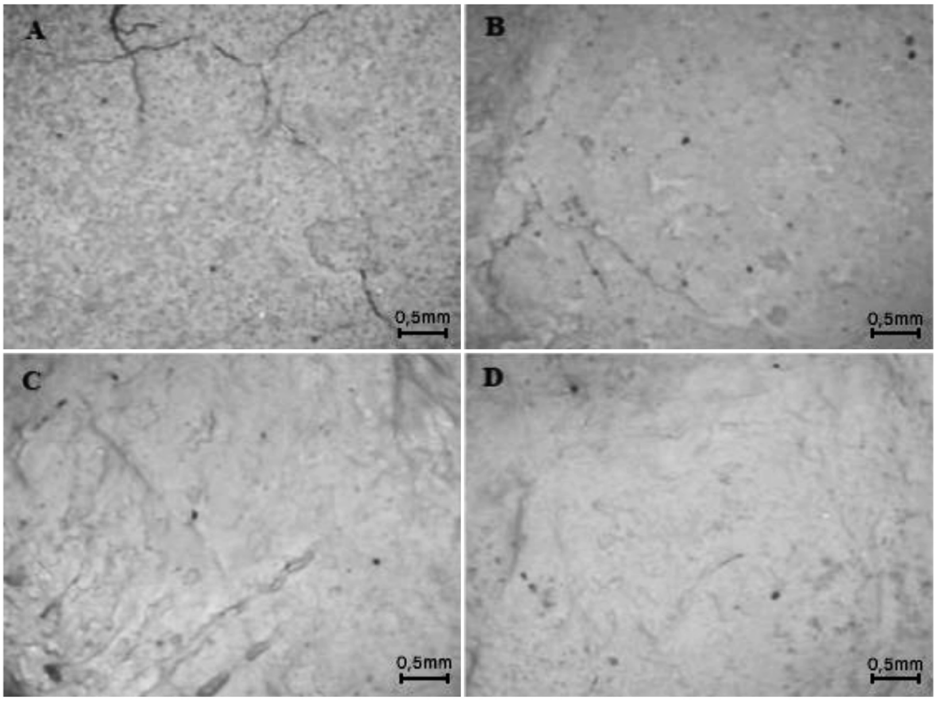

Fig. 1. The images of specimens surfaces made with Thymodentin material (A, B) and ZOE (C, D) after placing them for 28 days in artificial saliva solution at constant temperature of 36,6°C

Rys. 1. Obrazy powierzchni próbek wykonanych z preparatu Thymodentin (A, B) oraz tlenku cynku z eugenolem (C, D) po umieszczeniu ich na 28 dni w roztworze sztucznej śliny w stałej temperaturze 36,6°C

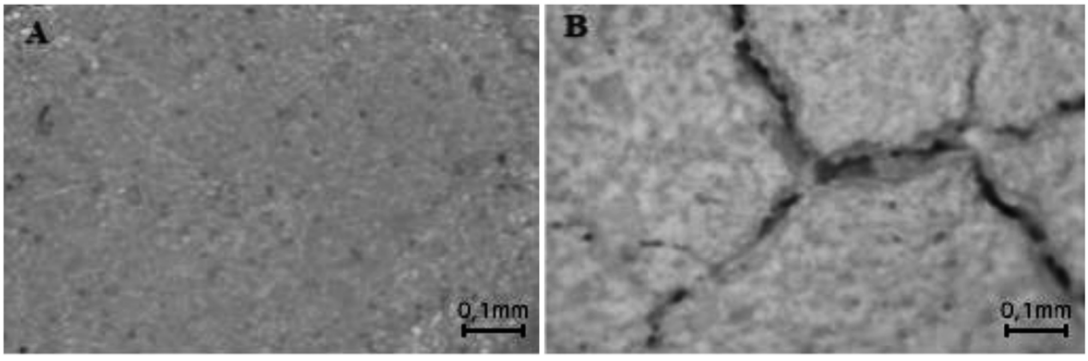

Fig. 2. The images of specimen surfaces made with Plastidentin material after its preparation (A) and after placing them for 7 days in artificial saliva solution at constant temperature of 36.6°C

Rys. 2. Obrazy powierzchni próbek wykonanych z preparatu Plastidentin po sporządzeniu próbki (A) oraz po jej umieszczeniu (B) na 7 dni w roztworze sztucznej śliny w stałej temperaturze 36,6°C

Table 4 contains the results of observation of changes in the specimens' surface after 7 and 28 days from their placement in artificial saliva solution imitating inflammation, stored at a constant temperature of 37°C. All specimens showed noticeable changes. The fastest reactions were observed in case of Plastidentin paste, where, after two days from the beginning of the test, the specimens dissolved. It may be the result of the reaction between the solution and material components. On the surfaces of other specimens, odds (except for the two specimens which were made from self-made Thymodentin paste), precipitation, and sediment were observed. These result from the rinsing and dissolution of the materials. **Figures 3** and **4** show, respectively, the surface layer changes with time of a specimen made from i-Pro cement and a specimen Thymodentin paste.

Table 4. The observation list results of surface structure changes of tested materials by organoleptic method after 7 and 28 days in artificial saliva solution imitating inflammation

Tabela 4. Zestawienie wyników obserwacji zmian struktur powierzchni badanych preparatów metodą organoleptyczną po 7 i 28 dniach w roztworze sztucznej śliny imitującej stan zapalny

Material	Time of tests	Organoleptic evaluation of the material
Thymodentin	7 days	– slight turbidity of the solution, – a precipitate settled, – visible odds appeared on the three specimens surfaces, – the surface of the other two specimens is smooth.
	28 days	– four specimens have been dissolved, – undissolved sample surface clearly undulate, – turbidity of the solution.
i-Pro	7 days	– appearance on the surface which had no contact with the glass, a white precipitate with a thickness of approx. 0.5 mm.
	28 days	– on the specimens surfaces appeared the knobbly precipitations, and the surface layer became clearly rough.
Plastidentin	7 days	– specimens dissolved 2 days after the beginning of the tests.
Zinc oxide eugenol (ZOE)	7 days	– the surface of the first specimen became undulate, small clusters of light-coloured precipitations appeared on it, as well as a small crack, – on the second specimen surface bright discolorations and white precipitate appeared, – on the other specimens deep cracks and clusters of coarse-crystalline precipitate were observed.
	28 days	– all specimens dissolved.

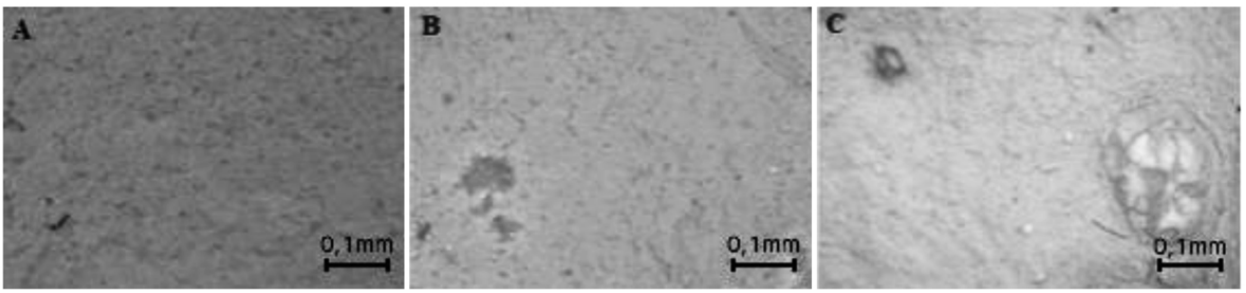

Fig. 3. The images of specimen surfaces made with i-Pro material after its preparation (A) and after placing them for 7 days (B) and 28 days (C) in artificial saliva solution imitating inflammation at constant temperature of 37°C

Rys. 3. Obrazy powierzchni próbek wykonanych z preparatu i-Pro po sporządzeniu próbki (A), po 7 dniach (B) oraz po 28 dniach badań (C) w roztworze sztucznej śliny imitującym stan zapalny w stałej temperaturze 37°C

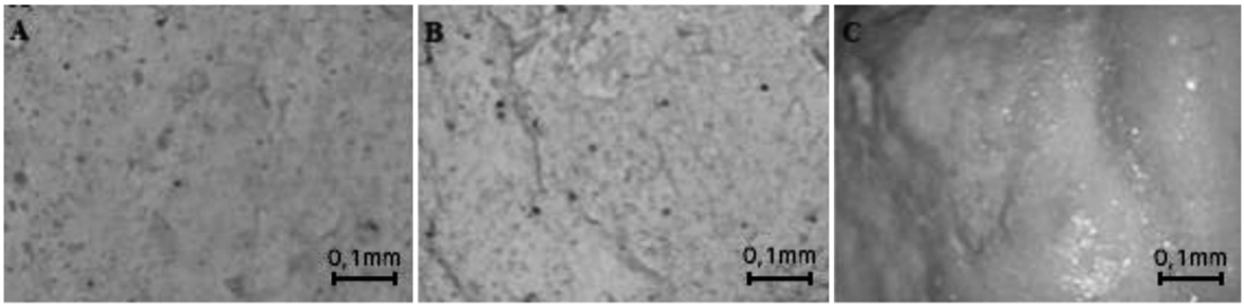

Fig. 4. The images of specimen surfaces made with Thymodentin material after its preparation (A) and after placing them for 7 days (B) and 28 days (C) in artificial saliva solution imitating inflammation at constant temperature of 37°C

Rys. 4. Obrazy powierzchni próbek wykonanych z preparatu Thymodentin po sporządzeniu próbki (A), po 7 dniach (B) oraz po 28 dniach badań (C) w roztworze sztucznej śliny imitującym stan zapalny w stałej temperaturze 37°C

Table 5 summarizes the average values of the percentage changes in the mass of specimens. The spread of results was within ± 2%. Additionally, to illustrate the intensity of changes shown in **Fig. 5**, the mass loss graphs are presented for specimens placed in artificial saliva solution at temperature of 36.6°C. The most noticeable differences appeared in case of specimens placed for 7 days in artificial saliva solution imitating inflammation at 37°C, especially for the laboratory-made Thymodentin paste, where the mass value has been reduced almost by half in relation to the initial value. The specimens made of Plastidentin cement dissolved after two days. One of the Thymodentin and all i-Pro specimens were the only ones that did not dissolve after 28 days of testing. The mass of specimens placed for 7 days in artificial saliva solution at temperature of 36.6°C decreased in a much lower range. Re-examination after 28 days showed a slight reduction of their mass. The temperature, the ingredients of solutions and preparations could significantly affect the dissolution of the tested materials.

Table 5. The percentage change in specimens mass for different time intervals

Tabela 5. Procentowa zmiana masy próbek dla różnych przedziałów czasowych

Material	The percentage change of mass			
	After 7 days in artificial saliva solution in temp. of 36,6°C	After 28 days in artificial saliva solution in temp. of 36,6°C	After 7 days in artificial saliva solution imitating inflammation in temp. of 37°C	After 28 days in artificial saliva solution imitating inflammation in temp. of 37°C
Thymodentin	22%	1%	44%	48%*
i-Pro	13%	3%	25%	24%
Plastidentin	6%	5%	–**	–**
Zinc oxide eugenol (ZOE)	4%	1%	20%	–***

* value for one specimen, the rest dissolved
** specimens were dissolved after 2 days from the beginning of the tests
*** specimens were dissolved

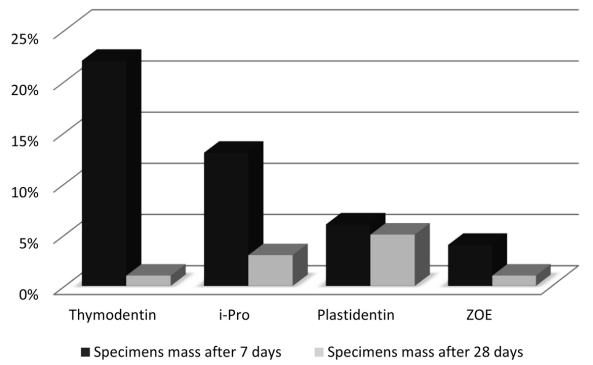

Fig. 5. The percentage mass loss of specimens under the action of artificial saliva solution at temperature of 36.6°C

Rys. 5. Procentowy ubytek masy próbek przy oddziaływaniu roztworu sztucznej śliny w temperaturze 36,6°C

Presented tests results showed significant changes in the surface structure of examined specimens. The dissolution of the material, precipitation, cracking and odds indicate degradation of materials within time.

Moreover, the temperature of 37°C (theoretically indicating the beginning of inflammation) and aggressive environment (simulation of inflammation) increase the dynamics of emerging changes. Tested material groups should demonstrate adequate marginal integrity and resistance to changes in the surface structure, due to purpose, for example, in the form of protection tooth cavity. Therefore, the loss of biofunctional properties under the influence of external factors may significantly affect the treatment under physiological conditions. In the treatment process, appearing loads should be included as a result from the process of chewing and opposing teeth pressure, which may lead to mechanical damage of used material. In the second phase of experimental studies, tribological tests were carried out and subjected to five specimens prepared for each of the analysed temporary filling materials. **Table 6** summarizes the average relative mass losses on the friction distance 5 and 10 meters, and **Fig. 6** presents the graph of relative mass loss. The spread of results was within ± 1.5%. Due to the different initial masses of specimens resulting from the method of specimen preparation (laboratory preperation and the different hygroscopicity of materials), a relative measure was taken as a measure of the wear process. The acceptance of the results enables one to compare the obtained results and to generalize observations and conclusions.

Table 6. Percentage mass loss of specimens for different friction distances

Tabela 6. Procentowy ubytek masy próbek dla różnych dróg tarcia

Material	Mass loss depending on the friction distance	
	5 m	10 m
Thymodentin	9%	5%
i-Pro	3%	1%
Plastidentin	5%	2%
Zinc oxide eugenol	6%	3%

Presented results show the mass changes of the specimens depending on the friction distance and varying intensity. The most crucial changes were observed for the Thymodentin material, and the least critical changes were observed for i-Pro cement. In case of tribological tests, the changes after a double increase of the friction path are more proportional than in the case of tests related to the influence of the environment (artificial saliva solution and imitating inflammation) after triple time increase. The examined materials also showed greater resistance to changes related to tribological wear than to changes caused by environment and temperature.

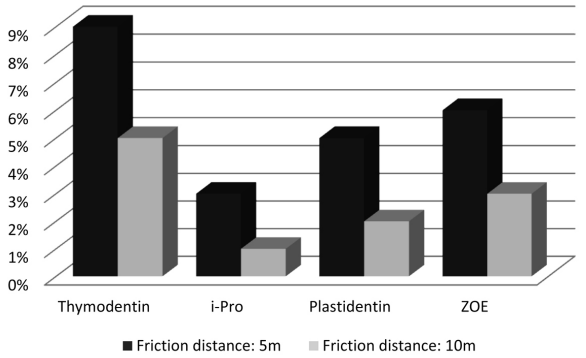

Fig. 6. Percentage mass loss of specimens for different friction distances

Rys. 6. Procentowy ubytek masy próbek dla różnych dróg tarcia

CONCLUSIONS

The aim of the conducted tests was an attempt to determine changes in the structure of temporary filling materials created under the influence of environmental conditions in time and tribological influence.

On the basis of the conducted tests, significant changes in the structure of the material were observed which arose in time under the influence of factors similar to the conditions prevailing in the oral cavity. Particularly, the reproduction of inflammation and elevated temperature caused the acceleration of the degradation of the material. Exceeding the recommended expiry date when filling of the defect may negatively affect the treatment. The observed changes in case of preparations could have arisen as a result of the selection of the amount of ingredients while making the paste as well as the time of binding the product in the solution environment. It was also noticed that the tested materials were characterized by higher resistance to abrasive wear than to the environmental impact.

Due to the fact that the conducted tests were preliminary and did not include such factors as variable pH of the oral cavity, the interaction of substances contained of beverages and food or variable force interaction in the chewing process, it would be beneficial to carry out studies taking into account these factors. Particularly, studies should be extended to tribological criteria. The friction process occurring in the stomatognathic system of a human often leads to an extinction of the treatment period.

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