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Test methods for adhesive polymer compositions in development and quality control for stoma care products on the example of adhesive baseplates (review)

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Abstract

Introduction. Adhesive baseplates, designed to fix ostomy bags and protect the skin of the peristomal area of ostomy patients, are a key element of an ostomy bag or urine bag. The functionality of adhesive baseplates is ensured by a set of their technical characteristics and design features, which makes the choice of appropriate baseplate testing methods an important aspect in the development, production and quality control of these medical devices.

Text. For testing adhesive baseplates, methods provided by Russian and international standards, as well as those not included in them, can be used. The methods provided by the standards include description of appearance (size), testing of adhesive strength, study of surface pH, as well as tests for wet integrity and water absorbancy. Methods not included in the standards for adhesive plates, but widely described in the scientific literature and used by researchers in practice include testing of some adhesive and mechanical properties, such as tack, shear strength, as well as tensile or bending tests to characterize the strength and deformation properties of the baseplate material. In addition, for adhesive baseplates, methods for testing medical properties common to all medical devices that are exposed to skin can be used. Besides this, it is shown how the adhesive properties of baseplates can be predicted based on the rheological properties of the adhesive layer, and factors important in the selection of materials modeling skin for *in vitro* tests are described.

Conclusion. The review presents a detailed description of methods of interest for testing adhesive baseplates, in particular their general principles and conditions of implementation, as well as methods that allow predicting the adhesive properties of plates and determining the relationship between adhesion tests *in vivo* and *in vitro*. A number of the described methods are of interest to include in normative documentation for adhesive baseplates tests, subject to their unification and modification taking into account the characteristics of this medical device.

Keywords: test methods, adhesive baseplate, medical devices, ostomy aids, adhesion prediction

Conflict of interest. The authors declare that they have no obvious and potential conflicts of interest related to the publication of this article.

Contribution of the authors. Ivan A. Sadkovskii conceptualised the study, collected and analized literature data and wrote the manuscript. Ludmila A. Korol, Anna B. Karabanova, Anastasia V. Korolenko collected and analized literature data and participated in the editing of the manuscript. Elena A. Shemetova, Ivan I. Krasnyuk, Galina V. Ramenskaya provided

consulting support. Mark A. Mandrik conceived the study idea, conceptualised the study, and participated in the writing of the manuscript. All authors participated in the critical discussion of the manuscript and approved the final version of the manuscript.

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Методы испытаний адгезивных полимерных композиций в разработке и контроле качества средств ухода за стомой на примере адгезивных пластин (обзор)

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Резюме

Введение. Адгезивные пластины, предназначенные для фиксации стомных мешков, а также защиты кожи перистомальной области стомированных пациентов, являются ключевым элементом калоприёмника или уроприёмника. Функциональность адгезивных пластин обеспечивается комплексом их технических характеристик и конструкционных особенностей, что делает выбор подходящих методов испытаний пластин важным аспектом в разработке, производстве и контроле качества этих медицинских изделий.

Текст. Для испытаний адгезивных пластин могут использоваться методы, как предусмотренные российскими и международными стандартами, так и не входящие в них. К предусмотренным стандартами методам испытаний относятся описание внешнего вида (размера), тестирование адгезионной прочности, исследование pH поверхности пластин, а также испытания на устойчивость к эрозии и абсорбционную способность пластин. Не включенные в стандарты на адгезивные пластины, но широко описанные в научной литературе и применяемые исследователями на практике методы включают в себя испытания некоторых адгезионных и механических характеристик, таких как липкость и прочность при сдвиге, а также испытания на растяжение или сгиб для характеристики прочностных и деформационных характеристик материала пластин. Также для адгезивных пластин могут применяться методы испытаний медицинских свойств, общие для всех медицинских изделий, контактирующих с кожей. Кроме того, показано, как адгезионные свойства пластин могут быть спрогнозированы на основе реологических свойств полимеров, входящих в состав адгезивного слоя, а также описаны факторы, важные при подборе материалов, моделирующих кожу в испытаниях *in vitro*.

Заключение. В обзоре представлена подробная характеристика методов, представляющих интерес для испытаний адгезивных пластин, в частности их общих принципов и условий проведения, а также методов, позволяющих прогнозировать адгезионные свойства пластин и определять связь между испытаниями адгезии *in vivo* и *in vitro*. Ряд описанных методов представляет интерес для включения в нормативную документацию на испытания адгезивных пластин при условии их унификации и модификации с учетом особенностей изделия.

Ключевые слова: методы испытаний, адгезивная пластина, медицинские изделия, средства для ухода за стомой, прогнозирование адгезии

Конфликт интересов. Авторы декларируют отсутствие явных и потенциальных конфликтов интересов, связанных с публикацией настоящей статьи.

Вклад авторов. И. А. Садковский – концепция работы, поиск и анализ литературы, написание текста обзора. Л. А. Король, А. Б. Карабанова, А. В. Короленко – поиск и анализ литературы, редактирование текста обзора. Е. А. Шеметова, И. И. Краснюк, Г. В. Раменская – консультационная помощь при написании данного обзора. М. А. Мандрик – автор идеи, концепция работы, написание текста обзора. Все авторы участвовали в критическом обсуждении результатов и утверждении окончательного текста обзора.

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INTRODUCTION

Ostomy is a common surgical procedure in the treatment of gastrointestinal diseases, such as colorectal cancer, inflammatory diseases, intestinal obstruction or perforation, as well as genitourinary diseases [1]. An intestinal stoma (ileostomy or colostomy) is formed by the removal of the end or loop of the intestine through the anterior abdominal wall, which makes it possible to preserve and ensure the excretory function of the intestine in case of its partial removal or the need for temporary isolation of certain areas (for example, anastomosis) [1, 2]. It is also common practice to use a fragment of the intestine to form a urostomy that serves to drain urine after radical cystectomy for genitourinary diseases, such as bladder cancer [3].

The stoma, as a rule, does not have a closure apparatus, so ostomy patients are not able to control the processes of defecation or urination [4]; In addition, caustic discharge from the stoma has a negative effect on the skin of the peristomal region and can cause skin erosion and contact dermatitis [5].

In this regard, the main practical aspect of stoma care is the use of stoma pouches or urostomy pouch – special devices worn on the body and designed to collect intestinal contents or urine and eliminate their aggressive effect on the skin (according to GOST R 58235-2022 "Assistive products for persons with excretory dysfunction. Terms and definitions. Classification").

A pouching system consists of a sealed container (stoma pouch) and an adhesive plate intended to fix the pouches hermetically and securely to the skin of the anterior abdominal wall around the stoma[1, 2, 3] [6–8]; The plate can be integrated into the structure (one-piece device) or connected to the pouch using an adhesive or mechanical flange connection (two-piece device) [6, 7]. Typical adhesive plates consist of a plastic substrate, such as polyurethane, laminated with one or more adhesive layers, and a protective release film that is removed immediately before the plate is applied. A flexible adhesive layer is typically a mixture (melt) of hydrophobic, pressure-sensitive adhesives (PSAs) and hydrophilic polymers that have hydrocolloid properties, and is essentially a thick composite film. In the center of the plate there is a hole intended for the stoma, which can often be modeled by cutting or by hand molding [6, 7].

The adhesive plate is a medical device that plays a key role in ensuring the functions of the pouch: in addition to attaching the ostomy pouch, it protects the skin of the peristomal area from stoma discharge and maintains the physiological functions of the skin.

[1] GOST R 58235-2022 "Assistive products for persons with excretory dysfunction. Terms and definitions. Classification". Available at: https://docs.cntd.ru/document/1200194434. Accessed: 24.07.2024.

[2] GOST R 58237-22 "Intestinal stoma care products: ostomy bags, assistive products and skin care products around the stoma. Characteristics and basic requirements. Test methods". Available at: https://protect.gost.ru/document.aspx?control=7&id=246873. Accessed: 24.07.2024.

[3] GOST R ISO 12505-1-2019. Skin barrier for ostomy aids. Test methods. Part 1. Size, surface pH and water-absorbency. Available at: https://docs.cntd.ru/document/1200166274. Accessed: 24.07.2024.

The effectiveness of the use of plates is largely related to the complex technical characteristics and design features of this product, which makes the selection of appropriate test methods for these characteristics challenging in quality control, as well as in the development and production of adhesive plates. Regulatory documentation for adhesive plate test methods is currently represented by the state standard GOST R 58237-2022 "Intestinal stoma care products: ostomy bags, assistive products and skin care products around the stoma. Characteristics and basic requirements. Test methods", as well as a series of standards GOST ISO 12505 under the general title "Skin barrier for ostomy aids. Test methods" consisting of two parts identical to the relevant international ISO standards. The standards include methods for testing the size, surface pH, water absorbency of the plates, and wet integrity and adhesive strength. However, they involve only a part of the characteristics that objectively affect the functionality of products, which determines the relevance of the review and other methods used to characterize adhesive plates.

Thus, the goal of this study is to characterize and analyze the current test methods for adhesive plates, as well as methods that are not regulated by regulatory documentation, but are of interest for testing plates and their materials.

Appearance

The design of adhesive plates plays an important role in ensuring their functionality. Different characteristics of appearance, such as shape, as well as linear and overall dimensions (length, width, diameter and thickness), directly affect the comfort of use and the effectiveness of peristomal skin protection for stomas of various types and sizes [4].

For example, in the case of a flat stoma, retracted stoma, or complex peristomal relief, flat adhesive plates provide less effective protection for the peristomal region than special convex plates, which can exert pressure on the skin around the stoma, thereby ensuring that it rises above the skin relief [4, 8].

The characteristics of the appearance of adhesive plates are provided by the regulatory documentation: the method is described in detail in GOST R 58237-2022 and GOST R ISO 12505-1-2019. The essence of the method is to determine the shape and dimensions (length, width or diameter), as well as the thickness of the adhesive plate. Based on the data obtained, the surface area of the plate is calculated, if necessary.

In addition, the diameter of the starting, ready, or modeled hole and flange, as well as the maximum diameter of the hole to be cut, are measured, if necessary.

According to the above standards, the shape of the adhesive plate is described according to the following characteristics given in Table 1.

In addition, the dimensions and thickness of the plate are measured using a measuring ruler and a thickness gauge.

The adhesive plates are measured according to their geometric shape according to the diagram shown in Figure 1.

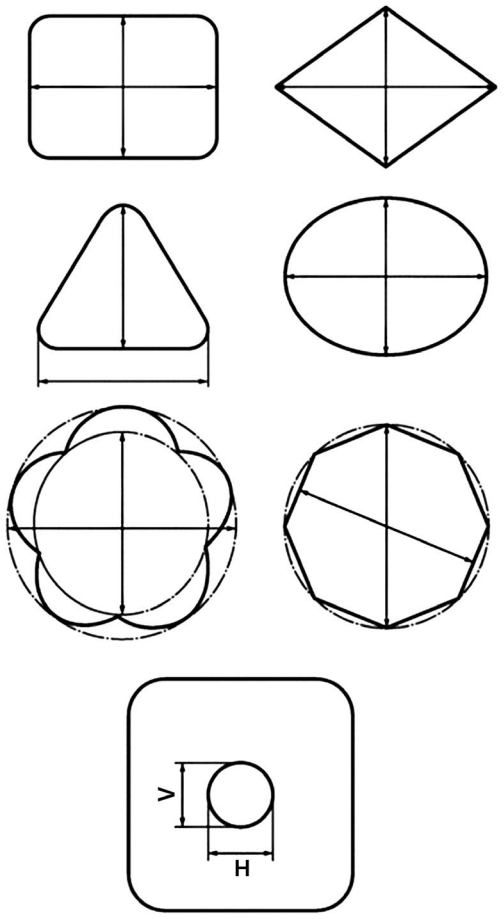

Figure 1. Scheme for measuring the dimensions of adhesive plates:
V – vertical length; H – horizontal length

Thickness measurements are taken in the center at the starting or prepared stoma opening, as well as at a distance of 5 mm from the outer edge. The plate thickness value is calculated as the difference between

the total thickness of the adhesive plate and the thickness of the protective film separately.

Table 1. Description of the shape of the adhesive plate according to normative documentation

Characteristics	Description
Skin barrier faceplate	Square, rectangle, diamond, triangle, circle, oval, others
Skin barrier cross section	Flat, convex, other
Skin barrier edge	Flat edge, tapered edge, others
Fringe of the skin barrier	No tape, tape border

Due to the fact that adhesive plates have different cross-sectional shapes, the standards establish several methods for measuring thickness, schematically presented in Figure 2.

It should be noted that for convex adhesive plates, one of the key appearance characteristics that determine functionality is also the steepness of convexity (dome slope), which in practice is quantified as the angle from the base of the plate to the top of the dome [9], however, the description of this aspect of the appearance of convex plates is not currently regulated by standards.

Mechanical properties

The mechanical properties of the adhesive hydrocolloid layer and polymer substrate are one of the main parameters that determine the technological and operational characteristics of adhesive plates. In particular, important conditions for ensuring the functionality of the product are sufficient strength to maintain integrity being exposed to externally, and at the same time sufficient flexibility to ensure the ability of the plate to adapt to body movements, maintaining close contact with the skin of the peristomal region in conditions of uneven relief and without causing discomfort.

At present, analytical procedures for evaluating the mechanical properties of adhesive plates are not available in current standards; however, there are a number of widely accepted standard methods used to study the mechanical properties of thin polymer films, in particular hydrocolloid wound dressings, transdermal as well as oral mucoadhesive films, making them potentially suitable for adhesive plates as well. These methods include tensile, puncture, folding, bending tests, etc. [10].

One of the main ways to characterize the mechanical properties of materials is tensile testing. In tensile testing of thin polymer films, in particular hydrocolloidal

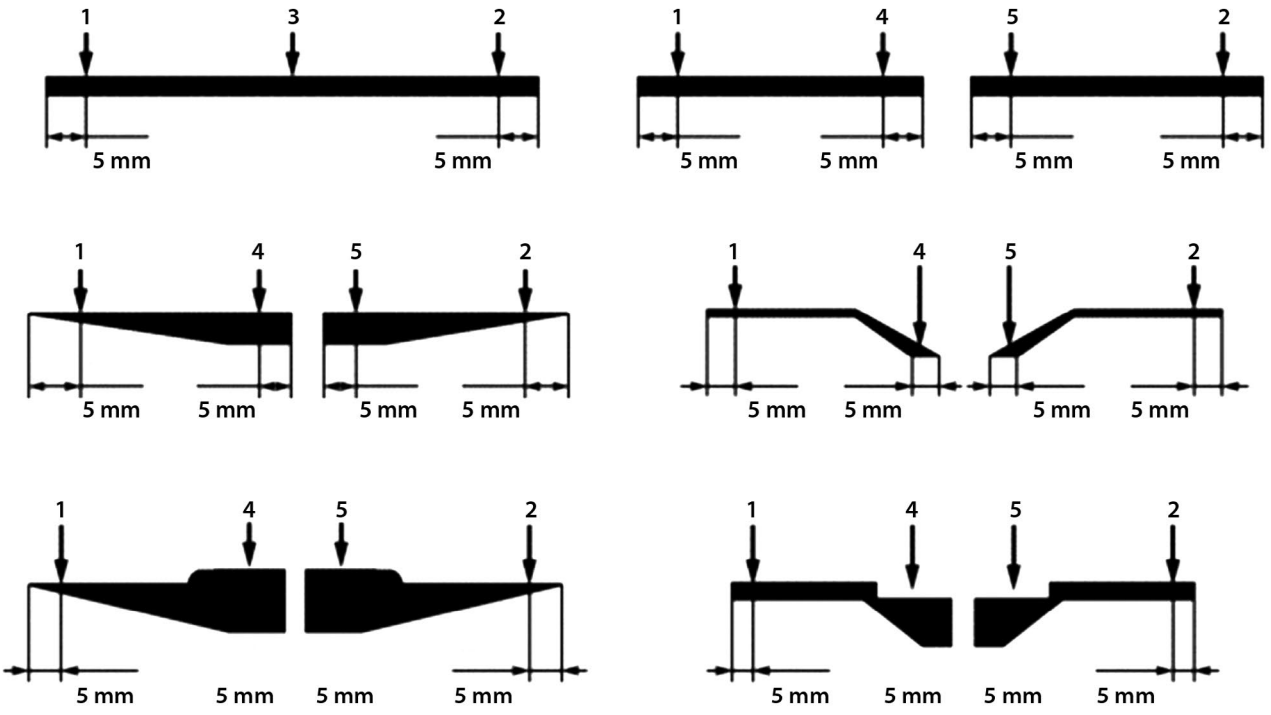

Figure 2. Scheme for measuring the thickness of adhesive plates:
1 and 2 – thickness measurement points on both sides along the periphery; 3, 4 and 5 – thickness measurement points in the center

films, methods based on the ASTM D 882 standard [11, 12], modified in terms of specimen size, have become common. The method consists of stretching a thin rectangular sample fixed in the clamps of a testing machine along its main longitudinal axis at a constant traverse speed. During the process, the tensile force withstood by the specimen, the force at which the specimen breaks and the specimen elongates relative to its original dimensions are recorded [11]. The strain-stress curve obtained from this test is used to calculate the tensile strength, modulus of elasticity, elongation, and tensile energy used as values of the strength, stiffness, plasticity and impact strength of the films, respectively [13].

The strength of the film can also be characterized in a puncture test. In this case, the resistance of the film to the compressive force is evaluated, which is carried out by fixing the sample horizontally using a special hollow device with the subsequent application of force perpendicular to the sample. This occurs by pressing the piercing probe on the surface of the specimen until it breaks, cracks, or the force that opposes the probe's movement disappears. Based on the results, a stress-strain curve is also obtained, which makes it possible to calculate puncture elongation and puncture energy [10].

Bending and folding tests can be used to characterize flexibility. The essence of the mandrel bending test, originally provided by ASTM D522/D522M17 to measure the flexibility of organic coatings on substrates, is to bend the specimen around an eight-millimeter cylindrical mandrel and then examine the specimen under a microscope for cracks, which characterizes the flexibility qualitatively [10]. The folding endurance test consists of repeatedly folding the sample by 180° in the same place until it breaks or tears. In this case, folding (bending) strength is expressed as the number of complete flexions [10, 13].

Adhesion properties

Adhesion properties are key characteristics of adhesive plates, critical to ensuring one of their main functions – fixation of stoma pouches or urostomy pouches on the skin for a long time.

These properties are determined both by the ability to form an adhesive bond (stickiness) and the ability to hold this bond being exposed to deformations of various kinds (adhesion and cohesion strength).

Stickiness is a property that characterizes the ability of a material to form a strong adhesive bond with the substrate when a small external pressure is applied to the adhesive for a short time (several seconds). It is the initial stickiness of the adhesive that provides the very possibility of forming an adhesive bond between the surface of the product and the substrate (skin or mucous membrane) [14].

Cohesion strength is understood as the intrinsic strength of an adhesive and is characterized primarily as the adhesive's resistance to tangential (shear) loads (e.g., during body movement or clothing friction). In this regard, the value of static shear resistance often determines the durability of the adhesive joint.

Adhesion strength can be indirectly characterized by peel resistance, which is the force required to break the adhesive bond when the adhesive peels off the substrate.

Since the removal of adhesive from the surface of the skin should be easy, without leaving a trace of a sticky layer, without causing pain or damaging the skin, the extent of peel resistance is the most important parameter in the development and quality control of adhesive plates [14, 15].

Testing of all of the above adhesion characteristics of plates is provided for by the JIS T 9233:1997 standard "Testing methods for ostomy aids" authored by the Industrial Standards Committee of Japan [16]. The Russian national standard GOST R 58237-2022, as well as GOST R ISO 12505-2-2019 "Skin barrier for ostomy aids. Test methods. Part 2" currently involves testing adhesive plates for adhesive strength only; At the same time, in accordance with the standard for the plate description, it is allowed to determine their other adhesive properties, such as stickiness and static shear resistance [17]. The adhesive strength test methods as well as the methods suitable for determining tack and static shear resistance are discussed in detail below.

Adhesive strength (peel test)

As a rule, to characterize the strength of the adhesive bond, the extent of force required to peel the adhesive off the substrate at an angle of 90° or 180° is used. However, it should be considered that the peeling activity involves not only the activity for breaking the adhesive bond, but also the activity required to stretch and bend the adhesive layer and substrate prior to breakage.

In this regard, with a large adhesive thickness and stiffness of the substrate, the peeling force can significantly exceed the true strength of the adhesive bond [18].

Nowadays, peel resistance tests typically use the equipment and procedures described in industry standards for thin self-adhesive tapes. These include the standards of the American testing and materials [ASTM D3330/D3330M04(2018), ASTM D6252/D6252M98(2019)], International Organization for Standardization (ISO29862:2018), European Adhesive Tape Association (AFERA 5001), etc.

The test consists of fixing the adhesive specimen on a steel test plate with the application of a certain measurement of the adhesive specimen separation force from the steel test plate when peeling at a specified angle (180° or 90°) and at a specified speed [14, 15]. The process is schematically shown in Figure 3.

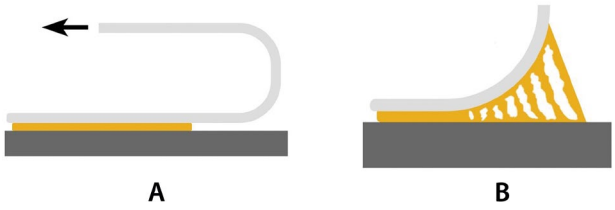

Figure 3. Peel adhesion test:
A – at 180°; B – at 90°

The 180° peel test from the metal substrate is a standardized test procedure for adhesive plates by JIS T 9233:1997[1], but its usefulness for these products is now questionable. In particular, this is due to the high values of the adhesive adhesion force of a thick adhesive plate to steel, which in practice leads to rapid stretching and rupture of the sample and does not allow you to obtain any realistic data.

Alternatively, a modified method was proposed, in which the substrate is a non-adhesive skin-like hydrophobic film made of fluorocarbon material that is peeled at an angle of 180° from the fixed adhesive plate sample; At the same time, the peeling speed is not the standard 300 mm/min, as provided for self-adhesive tapes, but 30 mm/min, which provides more stable measurement results [17]. In addition, this method eliminates the influence of substrate stiffness on the value of peeling adhesion [16].

This method of testing the adhesive strength of plates is currently provided for by the standards GOST R 58237-2022 and GOST R ISO 12505-2-2019[2].

Stickiness

Classical stickiness testing methods combine the study of the processes of formation and breakage of the adhesive bond. These include the rolling ball test, the probe tack test, and the loop tack test, all of which are described in the standards of the American Society for Testing and Materials. These methods are successfully used to study the stickiness of medical devices and PSA-based products, such as plasters and transdermal patches [15], and therefore are also of interest in the development and quality control of adhesive plates. In addition, the rolling ball method and a variant of the probe tack method known as the Mitsuhashi method are offered as official test methods for adhesive plates by JIS T 9233:1997.

When rolling ball method is used, the movement of a stainless-steel ball rolling down an inclined track is observed, which stops by touching an upward-facing adhesive layer at the bottom of the track. Stickiness is expressed through the distance traveled by the ball moving with a given initial momentum, controlled height and slope, until it comes to a complete stop. A short run length is equivalent to a high stickiness value, a long run length is equivalent to a low stickiness value [15].

The device shown in Figure 4 may be used for the test.

Detailed test conditions are given in the ASTM D3121-17 Standard Test Method for Tack of Pressure Sensitive Adhesives by Rolling Ball[3].

The method involves the study of the comparative tack of adhesives and is primarily intended for quality control, as it demonstrates good reproducibility within one laboratory and the ability to identify accurately changes from batch to batch, at a constant thickness of the adhesive layer. It is most suitable for adhesives with relatively low tack values, but is not recommended by this standard for the specification of final products [18].

Using the probe tack method, the test is carried out by measuring the force required to detach the probe

[1] JIS T 9233:1997. Testing methods for ostomy aids. Available at: https://kikakurui.com/t9/T9233-1997-01.html. Accessed: 02.08.2024.

[2] GOST R ISO "Skin barrier for ostomy aids. Test methods. Part 2. Wet integrity and adhesive strength". Available at: https://docs.cntd.ru/document/1200166275. Accessed: 24.07.2024.

[3] ASTM D3121-17. Standard Test Method for Tack of Pressure-Sensitive Adhesives by Rolling Ball. Available at: https://store.astm.org/d3121-17.html. Accessed: 02.08.2024.

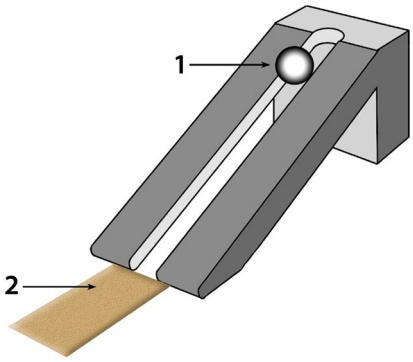

Figure 4. Rolling-ball device used to measure tack:
1 – stainless steel ball; 2 – sample

from the adhesive surface after applying low pressure. The method allows you to quantify tack as the maximum force required to break the adhesive bond after a short contact, as well as calculate a number of other characteristics of the bond, such as the axial rated stress (σ), maximum rated strain (εmax) and cohesion energy (Wadh), defined as the integral of the stress-strain curve (area under the curve) [15].

A texture analyzer equipped with a flat or spherical probe may be used to perform the test (Figure 5).

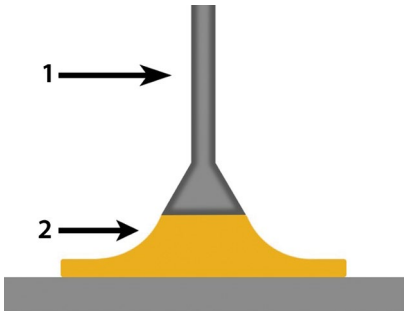

Figure 5. Probe tack device:
1 – flat probe; 2 – sample

The essence *of the loop method* is to determine the peeling force of the adhesive strip previously closed in the loop from the hard surface with which the loop is bonded by short-term contact with a small pressure, usually provided by the weight of the specimen itself. The test procedure is described in detail in the ASTM D619522 Standard Test Methods for Loop Tack. The method is designed to test adhesive masses and, according to the standard, it is suitable for both quality control and testing of adhesive properties[1] [15].

[1] ASTM D6195-22. Standard Test Methods for Loop Tack. Available at: https://store.astm.org/d6195-22.html. Accessed: 02.08.2024.

Shear resistance test

Static shear resistance testing of adhesives measures the force required to detach a standard area specimen from a standard flat surface (metal plate) in a direction parallel to the surface to which it is attached. This test quantifies the force required to displace the specimen on the plate by determining the time required to remove a standard area specimen from the plate when subjected to a standard constant load. A prerequisite for using the test results as a true measure of the intrinsic strength of the adhesive is the ccohesive type of the failure of the adhesive bond [14].

To perform testing of static shear resistance, the equipment schematically shown in Figure 6 is used.

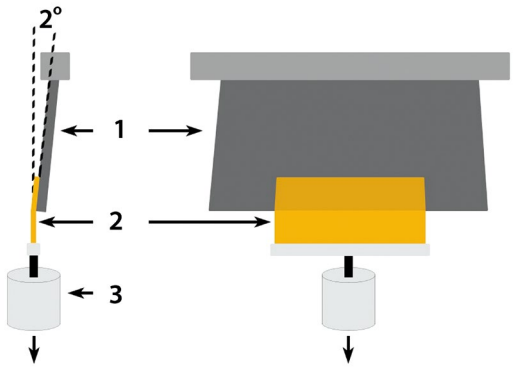

Figure 6. Shear test apparatus:
1 – test plate; 2 – sample; 3 – standard load

Detailed test conditions and methods are given in ASTM 3654/D3654M-06(2019) "Standard Test Methods for Shear Adhesion of Pressure Sensitive Tapes", ISO 29863:2018 "Self-adhesive tapes – Measurement of static shear adhesion", etc.

Determination of bioadhesion and prediction of adhesion in vivo

One of the main tasks in the development of adhesive plates is the assessment and prediction of their adhesion directly to the skin during operation. In this case, the standard adhesion tests performed on the skin of volunteers in vivo, described in a number of works [18, 19], although they allow obtaining the most relevant quantitative values of peeling or stickiness adhesion, are nevertheless not suitable for mass laboratory tests and product quality control due to the high variability of adhesion to the skin depending on gender and age, physiopathological status of the body, as well as the site of application [14]. In addition,

poor measurement performance is a problem, as well as certain security risks. An alternative may be the use of *animal skin ex vivo*, e.g., rat skin in the stickiness probing described in [20] for hydrocolloid dressings. This approach can be considered justified for the few adhesion studies at the product development stage, but even in this case, the problems of standardization of animal skin, as well as the performance and ethics of scaling up this test method, remain unresolved.

Standard methods for studying adhesion characteristics based on the interaction of the adhesive with a rigid metal substrate have a number of advantages, such as high performance, good reproducibility and relative simplicity. Nevertheless, the results of these measurements in practice often have a weak correlation with the adhesion of PSA to the skin, which is associated with significant differences in the surface and mechanical properties of the skin from metal substrates [14]. For example, clean, dry skin is a substrate with lower values of free surface energy (surface tension) than a standard steel plate, and therefore it is less wetted by the adhesive on contact, which leads to less initial tack [21]. In this regard, a number of investigators have proposed modifications of standard test methods, involving the replacement of high-energy metal substrates with low-energy polymeric substrates, such as polyethylene and polytetrafluoroethylene, in which the adhesion values were close to those for cadaveric skin [21, 22]. In particular, this approach was used to develop the already mentioned standard method for testing the adhesive strength of adhesive plates.

The critical surface tension of the skin is not a constant value; according to the literature, it increases as the relative humidity and temperature of the skin increase [22], which makes it difficult to use the above polymers to model the effect of humidity on adhesion *in vitro experiments*. For that, plates coated with collagen with different moisture content can be used as a substrate [23].

In addition to surface energy, adhesion to the skin is influenced by such parameters as roughness, as well as deformability (resistance) when being stretched and bent [14]. For example, a model material based on cross-linked gelatin proposed in the work by Lir et al. [24], which has the roughness of real living skin and comparable mechanical properties, demonstrated a lower adhesion strength in peel tests than a similar material with a smooth surface texture; At the same time, the values obtained for the rough material were very close to the results obtained for the skin. In addition, Renvoise et al. have produced a substrate the Young's modulus of which is close to the values typical of the skin. At a peel rate of 10 mm/min to 400 mm/min, such material deforms like skin and also exhibits a similar failure mode and similar peeling force values [25]. The above makes these synthetic materials a promising replacement for standard steel substrates in the study of the adhesion characteristics of drugs and medical devices, including adhesive plates.

Adhesion modeling based on rheological properties of adhesives

An important feature of adhesion parameters is the ability not only to measure them, but also to predict them with a high degree of accuracy based on the rheological characteristics of polymer adhesives.

As a rule, the elastomers that are part of the adhesive layer, when subjected to deformation, can demonstrate viscoelasticity – the properties of both liquids and solids. The experimentally measured values of dynamic stresses and dynamic deformation of a viscoelastic body subjected to sinusoidal oscillations make it possible to calculate their ratio – a value called the complex modulus G (for shear deformation).

The complex modulus is a quantity whose real part is the storage modulus (G'), characterizing the elastic component of the system's response, and whose imaginary part is the loss modulus (G"), corresponding to the viscous component of the system's response. The ratio between the loss and loss and storage modulus (G"/G') gives a value known as the loss tangent (tan δ), which is a measure of the amount of strain energy that is dissipated as heat during each stress cycle [14, 27].

At the moment, empirical criteria are known that make it possible to predict the manifestation of the stickiness of materials based on their rheological characteristics. The most widely known is the Dahlquist criterion, according to which stickiness occurs when the accumulation modulus for a polymer, determined at a frequency of 1 Hz, is less than 100 kPa [23]. More complex stickiness criteria are also used, in particular, Chang's "viscoelasticity windows" [27].

Shear adhesion tends to occur at slightly higher frequencies than initial tack and requires higher storage modulus values. At the same time, the value of static shear resistance is related to the plateau modulus on the frequency dependence curve of the modules the longer is the plateau of high elasticity, the higher is the shear resistance.

Finally, the process of adhesive joint failure when the adhesive is separated from the substrate occurs at high loading rates and requires a high cohesive strength close to a solid body. Therefore, high values of peel strength will be possessed by those PSAs for which the loss modulus prevails at low frequencies (G" > G'), and the elastic modulus prevails at high frequencies (G" < G').

Thus, for a for a typical elastomeric PSA, stickiness is manifested at low stress frequencies in the range of 0.005 to 0.05 rad/s, shear resistance, as a more high-speed process, is in the range of 0.05 to 0.5 rad/s, and peel adhesion, which is a typical high-speed process, occurs in the range of 100 to 1000 rad/s [26].

Dynamic Mechanical Analysis (DMA) is effective to determine the complex modulus and other rheological characteristics of viscoelastic hydrocolloid adhesives under high-frequency deformations. The method can be used in the testing of various materials in accordance with a number of standards, in particular ASTM D406520 "Standard Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures".

The essence of the DMA method is to study the viscoelastic properties of materials depending on time, temperature or frequency under various oscillating loads. This is achieved by sinusoidal deformation of the specimen followed by stress fixation: at a known stress, the specimen is deformed by a certain value, which depends on its stiffness. Therefore the method allows estimating the stiffness and damping of the test sample, for which the values of the modulus of elasticity and the loss angle are used, respectively.

In the development and study of adhesive plates, in addition to predicting the adhesion characteristics of the adhesive layer, the DMA method allows solving such problems as determining the viscoelastic range for polymers, studying the characteristics of polymer mixtures and the effect of modifying additives, aging of materials, the effect of the content of physically or chemically bound water on the properties of materials, changes in the properties of materials under the influence of environmental factors [18, 29].

Absorption of adhesive plates

The ability to absorb moisture from the surface of the skin of the peristomal region during prolonged wear is the most important property of adhesive plates, which contributes to the performance of one of the main functions of the plates – the protection of the peristomal skin, thereby preventing skin maceration and the development of pathogens [30]. In addition, the ability of the plate's hydrocolloidal components affects the adhesion values to the skin: when moisture is absorbed, adhesion increases, but excessive absorption will cause the adhesive to lose its tack.

The determination of water absorption for adhesive plates is provided in the regulatory documentation (GOST R 58237-2022 and GOST R ISO 12505-1-2019), according to which it is used to assess the sorption capacity of the adhesive (skin-contacted) surface of the adhesive plate using a cylinder with saline solution under test conditions for 6 hours. The test is based on the method for determining water absorption by wound dressings, provided for by the European standard EN 137261 2002 "Primary Wound Dressing Test Methods. Aspects of Absorbency".

The test procedure is as follows: Round specimens of adhesive plates with the protective coating removed are fixed to the test cylinders with an adhesive layer inside the cylinder. After weighing the plate cylinders and adding saline inside, the cylinders are temperature controlled at 37 °C for 6 hours. Then, having completely removed the residual liquid, the container with the sample is re-weighed.

The amount of liquid (K_v) absorbed by the plate is calculated from the difference in weight of the sample before and after contact with the liquid. Absorption capacity is expressed as the ratio of the amount of liquid absorbed to the area of the plate sample (mg/cm^2). The absorption test is shown in Figure 7.

Erosion resistance

The absorption of body moisture by the adhesive plates, as well as the possibility of contact with the discharge from the stoma, leads to their gradual deformation and over time to destruction (erosion), which leads to the loss of functionality of the plate. For this reason, one of the most important characteristics of adhesive plates that need to be tested in simulation of use is erosion resistance – the ability to maintain integrity when exposed to moisture and stoma secretions.

The test method for the wet integrity of plates, described in GOST R 58237-2022 and GOST R ISO 125052-2019, involves exposing the area around the central hole of a 25 mm diameter plate with saline solution during stirring, holding for 24 hours at 37 °C,

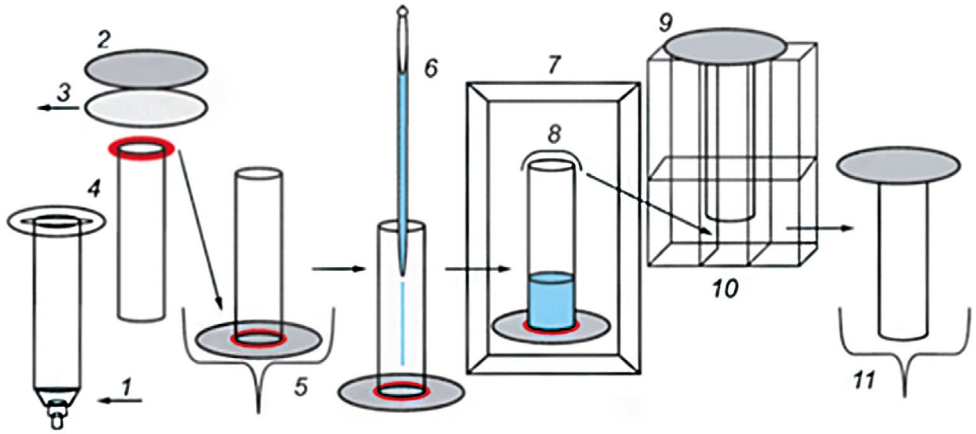

Figure 7. Scheme of the water absorption test:

1 – place of the syringe tip cut; 2 – adhesive plate; 3 – plate protective liner; 4 – syringe or cylinder with a fixing ring or flange; 5 – weighing of the cylinder before adding fluid; 6 – adding saline solution to the container; 7 – thermostat; 8 – film covering the cylinder; 9 – emptying the cylinder; 10 – stand for hanging the cylinder; 11 – weighing of the cylinder after the test

followed by measuring the inner diameter of the hole and the outer diameter of the plate. The results are described as follows: the inner diameter (holes) after the test of more than 25 mm is external erosion, less than 25 mm is internal swelling, the coincidence of the outer diameter of the plate and the edge of the discolored area is the collapsed boundary. It should be noted, however, that this test only takes into account the effect of moisture on the integrity of the plate and does not simulate the effects of aggressive enzymes and reactive solutes that are also present in the stoma discharge, which in turn can produce results that are significantly different from the conditions under which the plate is worn.

For the test, a device is used consisting of a test cup where saline is placed, a mounting disc to which a sample of the plate is attached, and a cover that is connected to the disc with a spacer tube and then closes the cup with the resulting structure (cover assembly). The device and the test procedure are shown in Figure 8.

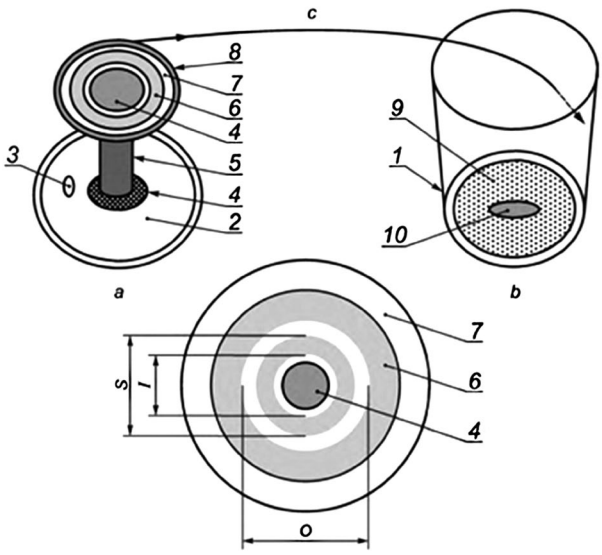

Figure 8. Device for testing the wet integrity of adhesive plates:

1 – test cup; 2 – cover; 3 – ventilation hole; 4 – clamps; 5 – fixing spacer tube; 6 – sample of an adhesive plate; 7 – waterproof plastic foam; 8 – mounting plate (disc); 9 – insulating material; 10 – magnetic stirrer. S – starting hole; I – internal diameter; O – outer diameter; a – cover assembly; b – cup; c – cover assembly in an inverted position

Vapor permeability

Vapor permeability is an important characteristic of adhesive systems designed to be worn on the skin for a long time, since the ability to transmit water vapor directly affects the adhesion of the adhesive layer to the skin and, consequently, the durability of the adhesive connection of hydrocolloid systems, diffusing through film, per unit area, time and pressure gradient [31].

Existing regulatory documentation for adhesive plate test methods does not provide for vapor permeability testing, but performing this test both on the entire plate and on the adhesive layer of the plate and substrate separately can be useful in understanding whether the product can maintain an optimal level of moisture

in the peristomal area. In this regard, it is possible to use general methods used to test a wide range of products and materials, such as hydrocolloid wound dressings, artificial skin, plastics and films, etc.

The most widely used method for measuring vapor permeability is the ASTM E96/E96M24 Standard Test Methods for Gravimetric Determination of Water Vapor Transmission Rate of Materials[1], also known as the cup method.

The method is based on the formation of difference in the vapour pressure of water on different sides of the test sample and gravimetric determination of the amount of water vapor that has passed through the unit area of the sample per unit of time. The test sample is positioned so that it tightly closes the neck of the test cup, inside which there is distilled water, which creates a certain vapor pressure depending on the temperature. A hermetically sealed cup is placed in a sealed chamber or desiccator with silica gel and stored at a constant temperature. The partial pressure gradient between both sides of the sample, due to the different relative humidity, creates a driving force that promotes the diffusion of water through the sample, resulting in a reduction in the weight of the cup. Under dynamic equilibrium conditions, the weight loss of the cup is constant, and the permeability of the sample at a given temperature is calculated from it [31].

Measurement of surface pH

Ensuring the optimal pH level of the surface of the adhesive plates plays an important role in preventing peristomal complications and ensuring comfortable wearing of stoma pouches (urostoma pouches). Both excessively high and excessively low pH values compared to the physiological pH of the skin can cause pain, skin irritation, and the dermatitis [32]. In this regard, the pH test of the plate surface plays an important role in the development and quality control of these products.

In accordance with GOST R 58237-2022 and GOST R ISO 12505-1-2019, the pH value of the adhesive surface of the adhesive plate in contact with the skin is measured in a moistened condition using a glass electrode pH meter with a flat electrode by pressing the electrode against the surface of the plate and fixing the pH value. The measurement is performed after 4 hours of contact of the adhesive plate sample with saline at a temperature of 37 °C and at least 1 hour at room temperature.

Medical properties

Irritant and sensitizing potential

Tests of the irritating and sensitizing potential of adhesive plates can be carried out in accordance with GOST ISO 10993-10-2011 “Medical devices. Biological evaluation of medical devices. Part 10. Tests for irritation and delayed-type hypersensitivity”. Tests are carried out *in vivo* (on animals and, in certain circumstances, on volunteers).

Microbial Purity

According to GOST R 53498-2019, microbial purity is determined according to GOST ISO 11737-1 “Sterilization of medical devices. Microbiological methods. Part 1. Estimation of population of microorganisms on products” for non-sterile medical devices of the adhesive type, except for fixing adhesive plasters that do not come into direct contact with the damaged surface of the skin.

CONCLUSION

The paper reviewed and analyzed the test methods for the properties of adhesive plates provided by the regulatory documentation, such as appearance, surface pH, water absorbency, erosion resistance and adhesion strength. In addition, methods that are not included in Russian standards, but are nevertheless of interest in the development and study of the technical and operational characteristics of adhesive plates, as well as in their quality control, including from the point of view of incorporating these methods in the relevant standards, subject to unification and adaptation to the features of adhesive plates, are described.

In addition to the standard-defined adhesion strength test, a number of methods can be used to study plate adhesion, such as the shear test and initial tack measurement, including the loop method, the rolling ball method, and the probe tack method. Besides, the possibilities of modifying standard methods for studying adhesion to increase the correlation between the test results and real adhesion to the skin are described. The possibility of using dynamic mechanical

[1] ASTM E96/E96M-24. Standard Test Methods for Gravimetric Determination of Water Vapor Transmission Rate of Materials. Available at: https://store.astm.org/e0096_e0096m-24.html. Accessed: 02.08.2024.

analysis to predict the adhesion characteristics of plates based on the rheological properties of the adhesive layer is also shown. In addition, possible methods for studying the mechanical characteristics of the components of adhesive plates, methods of determination of vapor permeability, as well as key medical characteristics of products are described and characterized.

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