An improved method of determining the viscosity of optical adhesives

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A method for determining the viscosity of adhesives used for bonding optical elements (hereafter called: optical adhesives) is presented in this paper. The Mitchel viscometer, which has been specially designed and made for the measurement of viscosity of adhesives, is used. The used appliance is brand new. The constant of viscometer (the constant of small ball) with the liquid of known viscosity-referential sample, has been determined. The oils of different viscosities, marked from RU-I to RU-VII, have been used as samples. Viscosities of the referential samples, used for this testing, have been previously specified according to the ANSI/ASTM D 445.

Key words: polymers, adhesives, optical adhesives, viscosity, viscosity measurement, testing method.

Introduction

PASTING optical elements comprises of filling gaps between optical elements with transparent liquid, adhesive, with the subsequent transformation of these liquid into transparent solid substance.

The surface tension of organic liquids is considerably lower than the free surface energy of inorganic glass and crystal, whereupon the optical adhesive, as a rule, moistens the bonding surface solidly. In order for an optical adhesive agent to quickly fill the gap between optical elements and squeeze out accidentally formed air bubbles, it is taken in surplus (the respective consumption of an adhesive is 5 to 20 ways higher in quantity).

The spreading of adhesives is developed according to the law of dynamics of the viscous incompressible liquids. Under the pressure of the upper element optical adhesive spreads, fills out the gap between glued surfaces and leaks on lateral surfaces. The rate of leaking of the optical adhesive along the surface is approximately equal to the rate of flowing adhesive in the gap thickness H, rapidly declines and is proportional to H^2 [1].

As the thickness of the layer gradually approaches the final value, the viscosity of optical adhesive grows. At that moment, operations of centering elements are performed. Adhesive leaks from the gap until the start of gelling. After the gelling process, the curing continues, and the slight approaching of glued surfaces occurs, due to the shrinking of adhesive. Since the adhesive is taken in the surplus, the final thickness of its layer practically does not depend from the start quantity of adhesive. It depends on the dimensions and shape of the glued elements, starting viscosity, rate of increase of viscosity and, in the end, on the force, that presses to the optical element which is glued. If the force is constant, the final thickness of the layer H (μm) can be calculated by the formula [2]:

$$H = F \cdot D^2 \cdot k \cdot \sqrt{\frac{\eta_0}{\tau_3 \cdot W}}$$

where

F – factor, depends on the shape of the glued surface,
D – dimensions of the glued surface, in mm,
K – coefficient, takes into consideration the shrinking of the adhesive after gelling,
η_0 – adhesive viscosity in the propagation start moment, in Pa·s,
τ_3 – time of enlargement of viscosity for e (2,73) times (time of adhesive gelling), in s,
W – force of gluing, in N.

According to the shape of glued surfaces, the D dimension can indicate:
- cycle diameter,
- ellipse axis,
- square side or
- width of rectangle.

The factor F brings out:
- for straight disc 0.38;
- for square 0.46;
- for ellipse $0.54 \cdot (E+E^3)^{-1/2}$;
- for rectangle $0.65 \cdot (E+E^3)^{-1/2}$, E is the ratio of the D dimension and the second ellipse axis length, or of the rectangle height;
- for spherical lentil area, limited by the circle, $0.38 \cdot \cos^2(\alpha/2)$, where $\alpha = \arcsin [D/(2R)]$ and R – radius curvature [3, 4].

The coefficient k is approximately one ($k<1$):

$$k = 1 - 0.01 \cdot Y$$

where
Y – the volume shrinking after gelling the adhesive, in %.

The rheological properties of adhesive η_0 and τ_3 can be determined from the curve which signifies the mutual dependence of adhesive viscosities and time. The force W, besides the weight of the upper element, can also include the additional force which is applied the glued elements (for example, weight of surcharge).

The requirements for the optical adhesives are:
1. the maximum light transparence and colorlessness in the set region of spectrum, purity of adhesive;

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2. index of refraction after the adhesive curing must be within the indices of refraction of glued materials;

3. the optic homogeneity provides for the absence of changes during the passage of light through the glued layer;

4. retaining of optical properties in the set temperature interval;

5. absence of residual stress in the adhesive layer after curing;

6. harmonization of the adhesive viscosity growth dynamics with the special pasting technology: protection of the start viscosity, bonding of elements without air bubbles, and getting the optimal thickness of the layer; maintaining viscosity analogous to the starting point, during the longer period (defined as the gelling time at 20°C); the growth of viscosity provided from the shear rate, which allows the execution of centering the optical elements;

7. the sufficient adhesive strength (cohesion) and sufficient joint strength (bond) with glued materials (adhesions) after curing, and retaining of their strength in an interval of set temperature and air humidity values;

8. the high adhesive elasticity and absence of visible plasticity;

9. retaining of the optical and mechanical properties of adhesives after curing in the long time.

Since the 1-6 requirements are specific, standard construction adhesives cannot satisfy them. Therefore the optical adhesives have been applied.

As experiments demonstrate, the optimal start viscosity of optical adhesives, depending on dimensions and shapes of glued optical elements, is usually in the range from 0.07 to 2.0 Pa·s. Since the viscosity of optical adhesives is directly proportional to the shear stress, the use of the viscometer by Mitchel is recommended for its measuring, since it works in conditions similar to the pasting conditions. The possibility of fast growth of viscosity is characteristic of every adhesive.

During curing, adhesive viscosity grows towards the exponential dependence, and it is suitable, because the gelling time at 20°C is long enough to execute the operation of final centering [5].

Viscosity

Viscosity is one of the essential attribute of polymeric solutions, (as optical adhesives are) that can be easily experimentally determined.

Viscosity is the friction force F, which emerges by parallel movements of two neighboring layers of a liquid of surface A, and is proportional to the gradient rate v along the axis y, normal to the direction of the movement of liquid:

$$F = \eta \cdot A \cdot \frac{\partial v}{\partial y}$$

The shear stress τ is proportional to the shear rate $\dot{\gamma}$:

$$\tau = \frac{F}{A} = \eta \cdot \frac{\partial v}{\partial y} = \eta \cdot \dot{\gamma}$$

The coefficient of proportionality η is called the coefficient to the viscosity (dynamic viscosity, absolute viscosity, or only viscosity). The unit for the dynamic viscosity in the SI has been the Pascal-seconds, (Pa·s)[2].

The relation between the dynamic viscosity η and the densities ρ of liquid is called the kinematic viscosity v, and is marked in the SI as square meters per second (m²·s⁻¹)[2]:

$$v = \frac{\eta}{\rho}$$

Measurement of viscosity is significant for the research of macromolecule. On the basis of measured data for the viscosity of the solution η and the viscosity of pure solvent η_0, the molar mass M and the geometry macromolecule, the special shape and the hardness of chain can be determined. For this the following values are used [8]:

The relative viscosity η_r presents the ratio of the viscosity of solution η and the viscosity of pure solvent η_0 measured under the same conditions:

$$\eta_r = \frac{\eta}{\eta_0}$$

The relative viscosity is the measure of change in the viscosity of solution compared to the viscosity of pure solvent, and it is usually greater than one.

The specific viscosity η_{sp} presents the quotient of the difference of viscosity of solution η and the viscosity of pure solvent η_0 with the viscosity pure solvent η_0, and represents the growth of viscosity above one:

$$\eta_{sp} = \frac{(\eta - \eta_0)}{\eta_0} = \eta_r - 1$$

The limiting viscosity number $[\eta]$, is defined with the expression:

$$[\eta] = \lim_{c \to 0} \frac{\eta_{sp}}{c} = \frac{\eta/\eta_0 - 1}{c} = \lim_{c \to 0} \frac{\eta - \eta_0}{c \cdot \eta_0},$$

and is used since the specific viscosity η_{sp} is a function of concentration, and is obtained through the extrapolation from graphic η_{sp}/c in a function from c for the value $c=0$.

The equation Mark – Kuhn – Houwink – Staudinger, which connects the limiting viscosity number $[\eta]$ with the molar mass M is appropriate for use:

$$[\eta] = K \cdot M^a$$

where K and a are empiric constants characteristic for the given polymer-solvent pair and the temperature. The constant a is a positive number and its value varies within the range from 0.5 to 1.0. The constants K and a are determined by an independent calibration methods through the use of samples of known molar mass. This equation enables parallel determining of molar mass on the basis of the limiting viscosity number measurements.

There are two procedures for the determining of liquid viscosity [9]:

- liquid is exposed to a constant force and the established shear rate is measured or

- liquid is exposed to a defined shear rate and the necessary force is measured.

Appliances which measure viscosity are called viscometers, and measuring is carried out during constant temperature and pressure. The most often used viscometers are:

- capillary viscometers, which measure the rate of the liquid flow through the pipe or the opening (by Ostwald,

[2] The unit in the International system unit.

by Ubbelohde),
- rotational viscometers, which measure the resistance which liquid gives under the influence of rotational element of appliance (by Brookfield),
- viscometers with the small ball, which measure time of the falling of a small ball through the liquid (by Hoeppler).

Viscosity of optical adhesives

The Mitchel viscometer handset has been introduced for determining optical adhesive viscosity. It has been made specially for this purpose in the laboratories of the Military Technical Institute in Belgrade.

Since the construction of the viscometer is brand new, the constant of viscometer is determined with the liquid of known viscosity - referential sample.

This must be done with all viscometers with a small ball, since there is not any other precise mathematical way to interpret the regime of flowing.

The testing appliance:
- The viscometer by Mitchel, presented in Fig.1, consists of a small steel ball /1/ of 25.400 mm in diameter, plastic handles /2/ and steel Č.4574 spherical body /3/.
- The spherical body /3/ has a slightly tilted spherical segment of 10 mm in diameter, fastened to the straight part of the handles /2/, which was predetermined for the setting of thermometer.
- The spherical body /3/ on its lower side has a part called the viscometer glass. It has been provided with three continuations set at an angle of 120°, of 1.8 mm in diameter, precisely polished off and hoisted 0.04 mm, so that between the surface of the small ball and the segment remains a gap of the same size.
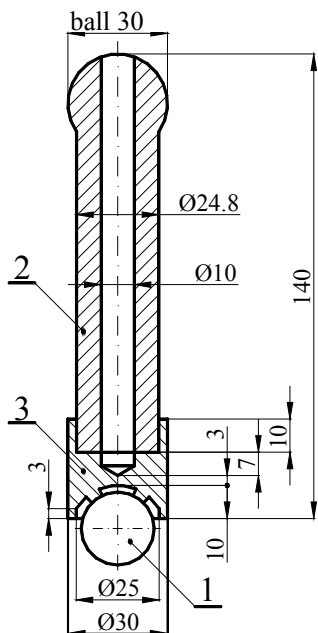

Figure 1. Viscometer by Mitchel

Standard viscometers with a small ball, for example that by Hoeppler, measure the time of falling of a small ball through the glass pipe, set at an angle of 80°, filled with liquid at a distance of 100 mm. While that by Mitchel measures the time of the fall of a small ball from the height of 70 mm, from the viscometer glass filled with liquid, the viscosity of which is determined. The total volume of examined liquid in by Hoeppler is around 40 cm³, while in

the case by Mitchel is less than 1 cm³.

By varying the densities (the steel), heights from which the small ball falls (70 mm) and the diameter of the small ball (25.400 mm) as well as the choice of geometry and dimension on the viscometer glass of the viscometer by Mitchel, viscosities of optical adhesives in the range from 0.07 to 2.0 Pa·s can be determined.

Owing to their construction, three continuations set in the viscometer glass ensure that the gap between the surface of the small ball and the segment is 0.04 mm. The size of this gap is especially significant for pasting a pair of lentils and all other optical elements.

Testing conditions:

Viscosity is determined in the dust-free room at a temperature of 20 ± 0.5^oC.

Viscometer must be calibrated with the liquid of known viscosity - referential sample.

Prior to the testing, the viscometer and sample adhesive must be adapted at a temperature of 20 ± 0.5^oC during 20 minutes.

Testing procedure:
- In the viscometer glass 5 to 6 drops of adhesive are poured, and the small ball is applied above. Adhesive must fill the entire gap between the small ball and the viscometer glass that must be visually checked. The viscometer with the small ball is turned for 180° and strongly pressed onto the desk, subsequently raised 70 mm above the table, while a stopwatch is activated. When the small ball falls on the desk the stopwatch is deactivated.
- Time difference in seconds, from the moment of lifting till the moment of falling of the small ball on the table, presents the time of the small ball fall by the sample of adhesive testing.
- Measurements on the viscometer are performed five times for determining an average value.
   Result calculation:
- The viscosity by Mitchel is calculated by the formula:

$$\eta_t = C \cdot \tau_t$$

where
η_t – the dynamic viscosity of the sample adhesive at 20^oC, in Pa·s,
C – the constant of viscometer or the constant of small ball, determined previously,
τ_t – the average arithmetic value of five measurements on the viscometer by Mitchel (the time of falling of the small ball by the testing of sample adhesive), in s.
- The results of the parallel testing of the same sample must not be greater than +5 % from the average value.
   Determining of the constant of viscometer C:
- In order to determine the constant of viscometer, at least five referential samples with known viscosities are prepared: 10 s, 20 s, 60 s, 120 s, 140 s and 180 s by Mitchel (see Table 1, column 2).
- Testing of every referential sample is performed ten times (recommendation: after every the third testing viscometers should be washed and dried before the testing continuation)
- The time of the fall of the small ball of every referential sample is taken as an average arithmetic value from the ten tests. The allowed deviation between two parallel tests must not be greater than 5% from the smallest value of the two results.
- After this, for all referential samples the kinematic viscosity, density and dynamic viscosity are determined

according to the standard method[3]. The dynamic viscosity of the referential sample equals kinematic multiplied with the density of the referential sample at a temperature of measurements. Only one testing is necessary for every referential sample.

- When the η_t and τ_t have been determined, the constant of viscometer C_i for the every referential sample is calculated along the formula:

$$C_i = \frac{\eta_t}{\tau_t}$$

where

C_i – the constant of viscometer of the referential sample,

η_t – the dynamic viscosity of the referential sample at 20°C, determined through the standard method, in Pa·s,

τ_t – the average arithmetic value obtained from ten measurements on the viscometer by Mitchel (the time of falling of the small ball by testing of the referential sample), in s.

- When the constants of viscometer C_i for each referential sample are determined, the constant of viscometer C is calculated by the formula:

$$C = \frac{\sum_{i=1}^{n} C_i}{n}$$

where

C – the constant of the viscometer by Mitchel,

C_i – the constant of viscometer of a referential sample,

n – number of referential samples.

Uncertainty of measurement of adhesive viscosity:

Uncertainty with which viscosities of adhesives can be determined is high. This depends on a few factors: kind of viscometer, uncertainty of readings of time, maintenance of constancy of temperature and on the purity of adhesives alone. Uncertainty measurement of viscosity of adhesives by Hoeppler is around ±2%, by Brookfield is 3% at most, by Ford is 2% at most [10], and uncertainty of measurement of viscosity of optical adhesives by Mitchel is 5% at most. When uncertainty of measuring is higher, testing must be repeated again.

Experimental

Testing is performed at a temperature of 20±0.5°C and at relative air humidity of 60 %.

For the calibration of the viscometer, oils of different viscosities have been used as referential samples, in the range from 0.08 to 1.70 Pa·s, of the following marks:

- HYDRAULIC OIL T-37 → RU-I
- HYDRAULIC OIL HV-46 → RU-II
- REFRIGERATOR OIL SHELL CLAVUS 68 → RU-III
- SINGLE GRADE TRANSMISSION OIL 90 → RU-IV
- MOTOR OIL №1 → RU-V
- MOTOR OIL №2 → RU-VI
- MOTOR OIL №3 → RU-VII

Necessary properties for all seven referential samples have been determined. Firstly, kinematic viscosities, in $m^2 \cdot s^{-1}$, and densities, in $kg \cdot m^{-3}$, and then dynamic

viscosities have been calculated[4], in Pa·s, towards the ANSI / ASTM D 445 [6]. The results are presented in Table 1, column 4.

On the viscometer by Mitchel the time of falling of the small ball, in s, has been measured, and then the constants of viscometer C_i for each referential sample have been calculated. The obtained results are provided as an average arithmetic value of 10 measurements (Table 1, column 3).

Firstly the constants of viscometer for each referential sample have been calculated (Table 1, column 5). The constant of viscometer C is calculated on the basis of values for constants of viscometer from all seven referential samples, as an average arithmetical value, and is 0.009932.

Table 1. Properties of referential samples necessary for determining the constant of viscometer

Mark referential sample	The know viscosity by Mitchel, in s	The measured viscosity by Mitchel, in s	Viscosity, towards the ANSI/ASTM D 445, in Pa·s	The constant of viscometer, C
1	2	3	4	5
RU-I	10	9.03	0.0861	0.009533
RU-II	10	10.25	0.0949	0.009256
RU-III	20	20.19	0.2286	0.011320
RU-IV	60	63.30	0.6897	0.010895
RU-V	120	120.77	1.0840	0.008975
RU-VI	140	138.34	1.4240	0.010293
RU-VII	180	182.81	1.6910	0.009250
The average arithmetic value:				0.009932

From the value of the constant of viscometer, the viscosity by Mitchel is calculated, in Pa·s, for these seven referential samples. The results are presented in Table 2, column 2.

Table 2. Values of viscosity of referential samples

Mark referential sample	Viscosity at 20°C, in Pa·s		The rel. error measurement, δ_{sr}, in %
	by Mitchel[5]	towards the ANSI/ASTM D 445[5]	
1	2	3	4
RU-I	0.0894	0.0861	1.9
RU-II	0.1022	0.0949	3.7
RU-III	0.2006	0.2286	6.5
RU-IV	0.6297	0.6897	4.5
RU-V	1.1998	1.0840	5.1
RU-VI	1.3925	1.4240	1.1
RU-VII	1.8126	1.6910	3.5

The values of viscosity from all seven tested referential samples practically show that the results match, although viscosities have been determined through different methods.

Therefore, in Table 2, column 4, the values and the relative error of measurements of viscosity from all seven referential samples for two different methods are presented, δ_{sr}, in %.

Relative error measurement values of viscosity of referential samples vary in the range from 1.1 to 6.5%. In six referential samples, the values of relative error are around and less than 5%, and only in one referential sample RU-III it is 6.5%. Relative error in measuring the values of viscosity of referential samples confirm that the differences between methods are very small, since uncertainty of

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[3] The ANSI / ASTM D 445 [6] .

[4] The dynamic viscosity (in Table: viscosity).
[5] Viscosity is determined: by Mitchel (method 1) and towards the ANSI/ASTM D 445 (method 2).

measurement of viscosity of liquid in the viscometer by Mitchel is 5% at most.

After calibrating the viscometer, the viscosities of examined adhesives have been determined. Two adhesives have been chosen, manufactured by LOCTITE EUROPA, and marked as:

- LOCTITE UV 350,
- LOCTITE UV 358.

They are single-component adhesives on the basis of urethane methacrylate, which cures under the influence of ultraviolet light (any gas lamp with had peak at a wave length from 365 nm, i.e. 0.365 μm can be used) in the duration of 20 to 30 s. Use for the glass bonding, because does not show any chemical reactions during contact with chemically sensitive glass; in literature data for their viscosities is stated: for the first adhesive from 3.00 to 6.00 Pa·s, and for the other one from 1.75 to 3.50 Pa·s [7].

For the viscometer by Mitchel, the time of falling of the small ball has been measured during the testing sample of adhesive, in s. The one result of tests is given as an average arithmetical value from 5 measurements.

Then the calculated viscosity by Mitchel, in Pa·s, and the results of testing of viscosity for these two adhesives are given in Table 3. For the sake of comparison, data from literature[6] for viscosities of these two adhesives have been presented in the same table.

Table 3. Review of viscosities of two adhesives LOCTITE UV 350 and UV 358

PROPERTY	Mark of adhesive sample	
	LOCTITE UV 350	LOCTITE UV 358
1. Viscosity by Mitchel at 20°C, η_t, in Pa·s	2.080	1.763
- the constant of viscometer, C	0.009932	0.009932
- time of falling of the small ball, τ_t, in s	209.9	177.6
2. Viscosity[6], in Pa·s	3.00 – 6.00	1.750 – 3.500

The viscosity of adhesive marked LOCTITE UV 358 is the concurrent with the data from literature. However, the obtained viscosity of adhesive marked LOCTITE UV 350 is not concurrent with the data from literature.

The measured values of viscosities of adhesives marked LOCTITE UV 350 and UV 358 are in agreement with theoretical values for the viscosity of optical adhesives, which is in the range from 0.07 to 2.0 Pa·s, which confirms that these two adhesives belong to the group of optical adhesives.

Conclusion

The method for determining the viscosity of optical adhesives on the viscometer by Mitchel has been developed. The method is simple for performance, and gives reliable testing results. For the verification of this method, two adhesives LOCTITE UV 350 and UV 358 have been tested, and in accordance with the obtained

values of viscosity, these two adhesives have been classified in the group of the optical adhesives.

This method, applied on the viscometer by Mitchel, can determine optimal starting viscosity of optical adhesives. It is especially important for the technology of gluing, which assures the bonding of elements without air bubbles, obtaining a layer of optimal thickness, and also the execution of centering optical elements.

In respect to the standard methods for the determination of adhesives viscosity, especially in optical adhesives, this method is special, since the consumption of the test sample which is of less than 5cm³ was able to define the quality and adequate choice of optical adhesives depending on dimensions and geometry of optical elements. It cannot be overlooked that optical elements are extremely expensive, and that construction of complex optical systems requires high precision.

The viscometer developed during the research presented in this paper, besides its basis purpose of optical adhesives testing, can be widely used for measuring viscosities of different liquids.

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Poboljšana metoda određivanja viskoznosti optičkih lepkova

U ovom radu prikazana je metoda za određivanje viskoznosti lepkova, koji se primenjuju za spajanje optičkih elemenata (u daljem tekstu: optički lepkovi). Korišćen je viskozimetar po Mitčelu, koji je projektovan i izrađen specijalno za merenje viskoznosti lepkova. Uređaj je potpuno nove konstrukcije. Određena je konstanta viskozimetra (konstanta kuglice) sa tečnošću poznate viskoznosti, referentni uzorak. Korišćeni su uzorci ulja različitih viskoznosti, oznaka RU-I do RU-VII. Viskoznosti referentnih uzoraka, korišćenih za ovo ispitivanje, prethodno su bili određeni prema ANSI/ASTM D 445.

Ključne reči: **polimeri, lepkovi, optički lepak, viskoznost, merenje viskoznosti, metoda ispitivanja.**

Улучший метод устанавливания вязкости оптических клеев

В настоящей работе показан метод для определения вязкости клеев, применявшихся для соединения оптических элементов (в дальнейшем тексте: оптические клеи). Тоже использован вискозиметр по Митчелу, спроектирован и изготовлен специально для измерений вязкости клеев и это оборудование совсем новой конструкции. Тоже определена постоянная вискозиметра (постоянная шарика) с жидкостью известной вязкости, референтный образец. Здесь использованы образцы масел различной вязкости, обозначений с РУ-I по РУ-VII. Вязкости референтных образцов, использованных для этого испытывания, предварительно были определены по АНСИ / АСТМ Д 445.

Ключевые слова: **полимеры, клеи, оптический клей, вязкость, измерение вязкости, метод испытывания.**

Méthode améliorée de la détermination de la viscosité des colles optiques

Dans ce papier on a présenté la méthode de la détermination de la viscosité des colles sont utilisées pour le liage des éléments optiques (dans le texte ci-après: les colles optiques). On a employé le viscosimètre de Mitchel, projeté et fabriqué spécialement pour le mesurage de la viscosité des colles. Ce dispositif a une construction complètement nouvelle. On a déterminé la constante du viscosimètre (constante de la petite boule) au moyen du liquide de viscosité connue, l'échantillon référentiel. Les échantillons des huiles de différentes viscosités, marqués par RU-I jusqu'à RU-VII ont été utilisés. Les viscosités des échantillons référentiels, employés dans cet essai, ont été auparavant déterminées selon ANSI/ASTM D 445.

Mots clés: **polymères, colles, colle optique, viscosité, mesurage de viscosité, méthode d'essai.**

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