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RESEARCH ARTICLE

N-[(1RS)-camphane-2-ylidene]aniline: A novel efficient liquid UV absorber for 3D printing

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Abstract

Objectives. To investigate the effectiveness of *N*-[(1RS)-camphanyl-2-ylidene]aniline as an ultraviolet (UV) absorber in 3D printing using digital light processing.

Methods. Polymerization process parameters were determined using a Netzsch DSC 204 F1 Phoenix differential scanning calorimeter equipped with an OmniCure S2000 UV irradiation attachment (400–500 nm filter). Samples were printed on a Minicube ULTRA 3D printer using a 405-nm LED light source. Dimensional accuracy during printing was evaluated according to ISO 52902:2019. Mechanical properties were determined using a Zwick/Roell Zwicki Z5.0 universal testing machine, while heat deflection temperature was measured on a Gotech HDT-HV-2000-3 device.

Results. The conversion degree of double bonds determined from differential scanning calorimetry results for a photopolymerizable composition containing camphor anil are almost identical to that for the composition without a UV absorber. The high gel fraction content in the samples indicates the formation of cross-linked polymers. The level of physical and mechanical properties, as determined in tensile and flexural parameters, is largely unaffected by the use of the type of UV absorbers considered. Tensile strength values are comparable to those of oligocarbonate methacrylate OCM-2-based materials produced under radiation polymerization conditions. Dimensional deviation for materials containing camphor anils is smaller than for compositions without a UV absorber or for compositions using a triazole derivative as an absorber.

Conclusions. The effectiveness of camphor anils as UV absorbers in the photopolymerizable composition is confirmed. With high dimensional accuracy in printing, it is possible to produce densely cross-linked polymers offering desirable physicomechanical properties and heat deflection temperatures.

Keywords

UV absorber, camphor anils, 3D printing, differential scanning calorimetry, oligocarbonate methacrylate

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НАУЧНАЯ СТАТЬЯ

N-[(1RS)-камфан-2-илиден]анилин — новый эффективный жидкий УФ-абсорбер для 3D-печати в условиях фотохимического инициирования

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Аннотация

Цели. Оценка эффективности применения *N*-[(1RS)-камфан-2-илиден]анилина в качестве ультрафиолетового (УФ) абсорбера при 3D-печати методом цифровой обработки света (digital light processing, DLP).

Методы. Параметры процесса полимеризации определялись на дифференциальном сканирующем калориметре Netzsch DSC 204 F1 Phoenix с приставкой УФ-облучения OmniCure S2000 (светофильтр 400–500 нм). Образцы печатали на 3D-принтере Minicube ULTRA со светодиодным источником излучения 405 нм. Линейную точность при печати оценивали по ГОСТ Р 59586-2021 (ISO 52902:2019). Прочностные характеристики определяли с использованием универсальной испытательной машины Zwick/Roell Zwicki Z5.0, деформационную теплостойкость — на приборе Gotech HDT-HV-2000-3.

Результаты. Значения степени превращения двойных связей, определенные по результатам дифференциальной сканирующей калориметрии для фотополимеризующейся композиции (ФПК), содержащей анилы камфоры, практически совпадают с таковыми для композиции без УФ-абсорбера. Высокое содержание гель-фракции в образцах свидетельствует о получении густосетчатых полимеров. Уровень достигаемых физико-механических свойств, определяемых показателями при растяжении и изгибе, практически не зависит от типа рассматриваемых УФ-абсорберов. Значения прочности при растяжении близки к характеристикам материалов на основе олигокарбонатметакрилата ОКМ-2, получаемым в условиях радиационной полимеризации. Отклонение от линейных размеров для материалов, содержащих анилы камфоры, меньше, чем при отсутствии в составе ФПК УФ-абсорбера или при использовании в качестве такового производного триазола.

Выводы. Подтверждена эффективность применения анилов камфоры в составе ФПК в качестве УФ-абсорбера. При высокой линейной точности печати реализуемо получение густосетчатых полимеров с высоким уровнем физико-механических характеристик и деформационной теплостойкости.

Ключевые слова

УФ-абсорбер, анилы камфоры, 3D-печать, дифференциальная сканирующая калориметрия, олигокарбонатметакрилат

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INTRODUCTION

Already widespread modern 3D printing technologies based on the use of oligomeric compositions that are reactive under photo-initiated polymerization conditions (VAT photopolymerization) [1–4] generally involve the use of a component or combination of components that act as a limiter of the polymer layer thickness during layer-by-layer build-up of the product under UV irradiation. Dyes and pigments used as such components [5–8] include those originally intended for the production of printed circuit boards [9, 10]. In addition, it is worth noting the presence of ultraviolet (UV) absorbers traditionally used in paints and varnishes, as well as in thermoplastics [1, 11, 12]. Since currently used components are not without drawbacks, the search for new effective UV absorbers for use in digital light processing (DLP) printing technology becomes a pressing issue. In particular, dyes can migrate into polymer optically transparent elements of printers that come into contact with photopolymerizable compositions (PPC), while pigments are prone to sedimentation settling during long-term printing. Industrial UV absorbers, which generally consist of powders of varying dispersion, have limited solubility in multicomponent oligomeric compositions.

The chemical structures of camphane- and aniline-based compounds on synthesized in previous works provided grounds to consider their potential applicability as UV absorbers for 3D printing [13]. Since there is no information of this kind in the scientific and technical literature, the present work set out to provide a comprehensive assessment of the efficiency of using one of the representatives of liquid camphor anils (*N*-[(1RS)-camphane-2-ylidene]aniline) as a UV absorber in PPC formulations.

MATERIALS AND METHODS

The model PPC consisted of oligocarbonate methacrylate OCM-2 (bis-(methacryloxyethylene carbonate)-diethyleneglycol, *Polyketone*, Russia), a bis-acylphosphine oxide (BAPO) (phenylbis(2,4,6-trimethylbenzoyl)-phosphine oxide photoinitiator, Omnirad 819, *IGM Resins*, Netherlands). (2-(2-hydroxy-5-methylphenyl)benzotriazole (*Tokyo Chemical Industry Co*., Japan), a triazole derivative (hereinafter triazole), was used as a comparative sample of the UV absorber. To determine the theoretical thermal effect, thermal polymerization was initiated with benzoyl peroxide purified by recrystallization (*Vekton*, Russia). Cyclohexane of chemically pure grade (*Vekton*, Russia) served as a solvent for UV spectroscopy. The experimentally determined concentrations of BAPO and UV absorbers that allow 3D printing with the same printer settings were 1.0 and 0.5% of the oligomer mass, respectively.

The PPC was obtained by mixing the ingredients. The mixtures were kept at 50°C for 1–3 h until the formation of homogeneous solutions. The characteristic parameters of the obtained PPC polymerization were determined in accordance with GOST R 56755-2015[1] (ISO 11357-5) on a DSC 204 F1 Phoenix differential scanning calorimeter (*Netzsch, Germany*) equipped with a compressor cooling system and an OmniCure S2000 UV irradiation attachment using a light filter transmitting in the 400–500 nm region at an irradiation power of 1 W/cm^2 in standard aluminum crucibles. The total flow of inert gas (argon) was 90 mL/min.

The theoretical thermal effect of polymerization, whose value was used to calculate the degree of conversion, was determined using differential scanning calorimetry (DSC) in dynamic mode (temperature scanning mode) by heating a sample of the OCM-2 oligomer containing dissolved benzoyl peroxide (0.5%) to 170°C at a rate of 5°C/min. The average of 5 measurements was 271.7 J/g, which is in fairly good agreement with the calculated data (271.0 J/g) based on the thermal effects known for methacrylates [14]. The thermal effects of photopolymerization were determined by performing two measurements equivalent in terms of sample exposure conditions and then subtracting the second measurement from the first. A similar calculation method was used in [15].

All samples were printed on a MiniCube ULTRA 3D printer (*Minicube*, Russia) equipped with a 405 nm LED light source. The preparation and slicing of print jobs was performed in specialized MiniCube Studio software. The specified layer thickness was 0.03 mm, while the number of layers, taking into account the supports, was 2375. After printing, washing with isopropanol, and mechanical removal of supports, the samples were dried at room temperature for 30 min. Post-polymerization was carried out at 60°C for 60 min on each side on a FormCure installation (*Formlabs*, USA) equipped with LEDs having an emission wavelength of 405 nm. UV spectra were recorded on a UV-2600 device (*Shimadzu*, Japan) in standard quartz cuvettes with an optical path length of 10 mm.

The intensity of the 3D printer LED source at the bottom of a standard container was measured using a UVpad E UV radiometer (*Opsytec Dr. Gröbel*, Germany).

Linear accuracy during printing was assessed according to GOST R 59586-2021[2] (ISO 52902:2019).

To determine the amount of gel fraction, extraction with boiling toluene was carried out for 24 h using a Soxhlet apparatus.

The strength characteristics of the samples were determined using a Zwick Z5.0 universal testing machine (*Zwick/Roell*, Germany) in accordance with GOST 34370-2017[3] (ISO 527-1:2012): a sample of

[1] GOST R 56755-2015. National Standard of the Russian Federation. Plastics. Differential scanning calorimetry (DSC). Part 5. Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion. Moscow: Standartinform; 2016 (in Russ.).

[2] GOST R 59586-2021. National Standard of the Russian Federation. Additive technologies. Test artifacts. Geometric capability assessment of additive manufacturing systems. Moscow: Russian Institute of Standardization; 2016 (in Russ.).

[3] GOST 34370-2017. Interstate Standard. Plastics. Determination of tensile properties. Part 1: General principles. Moscow: Standartinform; 2018 (in Russ.).

type 4 (GOST 11262-2017[4] (ISO 527-2:2012)) was characterized at a speed of 100 mm/min, while a sample of 80 × 10 × 4 mm bar type (GOST 4648-2014[5] (ISO 178:2010)) was tested at a speed of 2 mm/min.

Heat deflection temperature (HDT) was determined in accordance with GOST 12021-2017[6] (ISO 75-2:2013) using a Gotech HDT-HV-2000-3 (*Gotech Inc*., China) device. The samples were comprised of 80 × 10 × 4 mm bar. The heating rate was 2°C/min, while bending stresses were 0.45 and 1.8 MPa, respectively. Shore D hardness was measured in accordance with GOST 24621-2015[7] (ISO 868:2003) using a TH210 portable hardness tester (*Time Group Inc*., China).

RESULTS AND DISCUSSION

Previously [16], we used a detailed visual comparison to demonstrate the possibility of using camphor anils as UV absorbers for 3D printing. As such, it was deemed necessary to numerically evaluate not only the detailing during printing, but also the complex of properties of the resulting materials. The motivation for choosing OCM-2 as the base oligomer consisted in the possibility of 3D printing samples without any additional components, such as adhesion modifiers or wetting modifiers. Detailed information, including information on the synthesis, properties of OCM-2 and the physical and mechanical characteristics of polymers based thereupon, is given in [17, 18].

The DSC method was used to obtain sets of values of the total enthalpies of reactions, whose averaged values were then used to calculate the degree of conversion. The results are presented in Table 1.

Table 1. Enthalpy of reaction and conversion degree of the photopolymerizable composition at 20°C

UV absorber	Total enthalpy of reaction, J/g	Degree of conversion, %
Without absorber	220.9	81.0
Triazole	206.3	75.9
Camphor anil	219.5	80.8

It can be seen from the obtained data that the degree of conversion at 20°C does not reach 90% in all cases. However, the values for the PPC-containing camphor anils practically coincide with those for the composition without a UV absorber. Increasing the temperature to values created in the printing zone (30–40°C depending on the device) should promote a deeper polymerization, which is demonstrated by the values obtained when processing the results of experiments conducted at different temperatures (Table 2).

Table 2. Conversion degree of the photopolymerizable composition at different temperatures

Temperature, °C	Conversion degree, %	
	Without UV absorbers	Camphor anil
20	81.0	80.8
30	91.1	93.1
40	95.9	93.5

Considering that the post-processing of the samples includes not only the rapid removal of unpolymerized PPC residues from the samples with isopropanol and drying, but also the stage of post-polymerization at an elevated temperature, it was assumed that the final degree of conversion of the oligomer into a polymer would be higher than that calculated from the DSC results. The degree of curing achieved under real conditions (3D printing with subsequent post-processing) was determined using gel-sol analysis. The content of the gel fraction in the samples, which was 98.2–98.3%, indicates the production of densely networked polymers and the practical absence of a significant effect of UV absorbers in the studied concentration on the formation of three-dimensionally cross-linked products.

An assessment of the physical and mechanical characteristics obtained under 3D printing conditions of materials (Tables 3 and 4) showed that the level of achieved properties determined by the indicators under tension and bending is practically independent of the UV absorber used; moreover, the difference in values is within the limits of measurement error.

[4] GOST 11262-2017. Interstate Standard. Plastics. Tensile test method. Moscow: Standartinform; 2018 (in Russ.).

[5] GOST 4648-2014. Interstate Standard. Plastics. Method of static bending test. Moscow: Standartinform; 2016 (in Russ.).

[6] GOST 12021-2017. Interstate Standard. Plastics and ebonite. Method for determination of temperature of deflection under load. Moscow: Standartinform; 2018 (in Russ.).

[7] GOST 24621-2015. Interstate Standard. Plastics and ebonite. Determination of indentation hardness by means of a durometer (Shore hardness). Moscow: Standartinform; 2018 (in Russ.).

Table 3. Properties of materials under bending stress conditions

UV absorber	Modulus of elasticity, GPa	Maximum force, MPa	Relative elongation at maximum force, %	Force at failure, MPa	Relative elongation at failure, %
Without absorber	3.06	116.2	5.9	112.4	7.0
Triazole	3.09	117.6	6.1	114.2	7.0
Camphor anil	3.10	117.4	5.7	115.6	6.0

Table 4. Properties of materials under tensile stress conditions and Shore D hardness

UV absorber	Tensile strength, MPa	Relative elongation, %	Shore D hardness, conventional units
Without absorber	79.0	4.8	89
Triazole	80.4	5.5	89
Camphor anil	78.4	4.4	89

It should be noted that the obtained tensile strength values are 20% higher than similar values for samples formed under conditions of material initiation, as well as being close in values to materials obtained under conditions of radiation polymerization [17]. Apparently, this is due to the absence of a temperature gradient during polymerization realized under the conditions of the 3D printing method used at the selected layer thickness and the consequent minimization of internal stress.

The differences in the values of heat deflection for different component compositions of PPC (Table 5) are within the limits of experimental error.

Table 5. Heat deflection temperature

UV absorber	Bending stress, MPa	T, °C
Without absorber	0.45	91
	1.8	72
Triazole	0.45	88
	1.8	69
Camphor anil	0.45	90
	1.8	69

Linear accuracy during printing, which is of decisive importance in additive technologies, was assessed using a standard sample (Fig. 1).

The numerical values given in Table 5 confirm the possibility of using camphor anil to obtain samples having a smaller deviation from the linear dimensions than in the absence of a UV absorber in the PPC composition or when using triazole as such. In general, the deviation of the linear accuracy does not exceed 1% (Table 6).

It should be noted that the absorption of anil camphor in the UV range (Fig. 2) in the region of maximum emission (406–409 nm) changes insignificantly (from 0.042 to 0.036), whereas in case of triazole it decreases more than twofold (from 0.056 to 0.024). Apparently, this is the reason for the greater linear accuracy when printing using anil camphor as a UV absorber.

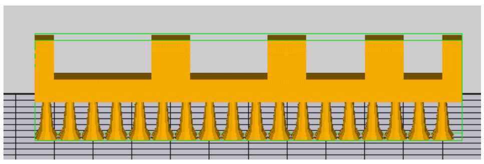

Fig. 1. Sample model for determining linear accuracy

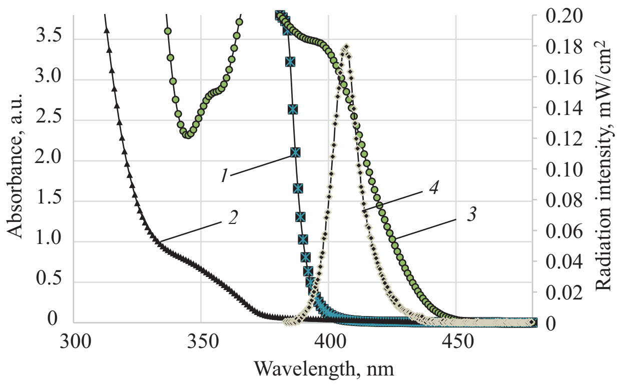

Fig. 2. UV spectra of triazole (*1*), camphor anil (*2*), and photoinitiator BAPO (*3*); emission spectrum of the 3D printer radiation source (*4*)

Table 6. Results of linear accuracy determination during printing

Specified size, mm	Deviation from specified size, %		
	Without absorber	Triazole	Camphor anil
Block dimensions			
55	1.22	1.29	1.00
5	0.93	1.00	0.09
8	1.11	1.42	0.64
Distance between tabs			
5	−0.07	−0.11	−0.16
7.5	−0.05	−0.09	−0.05
10	−0.04	−0.05	−0.04
12.5	−0.09	−0.20	−0.04
End tabs			
2.5	0.05	0.24	0.18
5	0.15	0.22	0.18
5	0.24	0.22	0.15
5	0.47	0.24	0.07
2.5	0.16	0.20	0.09

CONCLUSIONS

Thus, the analysis of the totality of the obtained results confirms the efficiency of using camphor anil in the PPC composition as a UV absorber. With high linear printing accuracy, it is possible to obtain densely meshed polymers characterized by desirable physicomechanical characteristics and heat deflection temperature.

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Authors’ contributions

N.V. Sidorenko—research concept, DSC measurements, initial manuscript drafting.

M.A. Vaniev—adjustment of the experimental plan, final manuscript preparation.

Iu.M. Mkrtchyan—3D printing, post-processing, property determination, and linear accuracy determination.

N.A. Salykin—synthesis of camphor anil, preparation of photocurable composition, gel–sol analysis, DSC data processing.

A.A. Vernigora—extraction and purification of camphor anil.

I.A. Novakov—adjustment of the experimental plan, final manuscript preparation.

The authors declare no conflicts of interest.

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