



Aluminium oxide particles-containing organogels for cleaning of optical glass surfaces

Galina Vladimirovna LYAMINA¹, Oksana Valerievna DUBININA^{1,*}, and Alfa Edison ILELA¹

¹ Tomsk Polytechnic University, Division for Material Science, School of Advanced Manufacturing Technologies, Lenina Avenue 30, Tomsk, 634050, Russia

*Corresponding author e-mail: dubininaov@tpu.ru

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Abstract

Hypothesis:

Some types of cleaning methods are not very effective and can actually increase damage to the optical parts being cleaned. As an alternative to these methods, we propose the use of gel-like substances to remove contaminants from optical surfaces. We have developed a method for cleaning optical glass surfaces that uses organogels, which are based on copolymers of methyl methacrylate and methacrylic acid. These organogels are loaded with polyethylene glycol, and aluminum oxide particles (Al_2O_3), which help to effectively remove contaminants without causing damage to the surface.

Experiments:

Al_2O_3 nanoparticles prepared by a Nano Spray Dryer were added to monomer stock solutions, and free-radical polymerization was conducted. Light crowns, heavy phosphate crowns and flints were used as research objects. Solutions of indene-coumarone resin in toluene and nitrocellulose enamel were used as contaminants. Polymer gels were characterized using resistance measurements, the gravimetric method in various solvents, and infrared spectroscopy. Optical microscopy and Atomic Force microscopy were used to observe changes on the glass surfaces.

Findings:

The advantages of this method include: effectiveness, simplicity, the use of small volumes of liquid solvents and aggressive media, and the ability to clean objects with any curvature or relief. The addition of Al_2O_3 particles as a reinforcing filler to the gel has made it easier to remove films from cleaned optical glass surfaces.

1. Introduction

The manufacturing of optical parts for high-precision instruments and observation systems involves a variety of operations, including cutting, grinding, and polishing [1,2]. They are also cleaned with various solvents [3-6], and exposed to physical fields such as hydrogen radicals [7] and laser plasma [8]. It is important to note that the use of grinding powders, polishing pastes, and solvents can cause defects in the optical surfaces of these components, such as cracks and scratches.

Additionally, the contaminants from the atmosphere or after a contact with humans, accumulate on the optical part surfaces during the operation [9,10], making cleaning more difficult and necessitating mobile cleaning methods. This is especially true when optical components are connected to equipment and cannot be cleaned with a solvent. Traditionally, the standard cleaning process involves removing contaminants by wiping the optics. However, this approach is not very efficient and can significantly damage the optical surface.

It is known that removing contaminants from optical surfaces is a common, delicate, and complicated task [11]. Sometimes the alternative methods are used, for example, application of gel-like substances [12-16]. The advantages of these methods are quality, simplicity, the use of small volumes of liquid solvents, and the low probability of

damaging the processed optical parts. Such substances can be used to clean optical parts of any curvature or relief. Importantly, there are many options available for cleaning optical objects today, for example, wipes impregnated with polymer-based cleaning solution, polymer films, pens and etc.

Chemical reactions in gels differ significantly from those in solutions. On the one hand, processes in gels are generally slow because cross-links in the polymers hinder diffusion. On the other hand, reverse reactions are difficult because of strong cooperative effects and solvation between functional groups in polymers and low-molecular-weight components [17-19].

In our previous work, we demonstrated the application of organogels to remove contaminants from metal surfaces. To improve cleaning efficiency, we introduced alkali metal trifluoroacetate, being enough toxic substances, into the gel matrix [17,20].

In this work, we present a method for cleaning optical glass surfaces using organogels based on copolymers of methyl methacrylate and methacrylic acid, loaded with polyethylene glycol, and aluminum oxide particles. The main idea is to preserve the functional properties of the polymeric matrix while replacing toxic substances with chemically inert particles. This approach allows us to combine the advantages of processes into gels with an eco-friendly approach to surface cleaning.

Aluminum oxide particles are chosen as an inert material because the technique for their synthesis using a Nano Spray Dryer is well-developed [21].

2. Experimental

2.1 Materials

For the synthesis of the gels, the reagents: methylmethacrylate (MMA), methacrylic acid (MAA), polyethylene glycol 200 (PEG) and benzoyl peroxide were purchased from Merck (Germany) and Sigma-Aldrich (USA), respectively. To prepare the polymer gel films and aluminum oxide nanopowder, the reagents: toluene (Tol), butyl acetate (BA), methyl cellosolve (MC) and aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$) were purchased from Ekos 1 (Russia) and Reagent (Russia), respectively. The contaminants of the optical glasses: indene-coumarone resin and nitrocellulose enamel were purchased from Ruskhimset (Russia). All the reagents were used as received without any purification.

2.2 Synthesis of aluminum oxide nanopowder

The preparation of aluminum oxide powder was carried out by the nano-spray drying method (Nano Spray Dryer B-90, BUCHI, Switzerland), using a 0.25 M aluminum sulfate suspension in distilled water as the starting material. The following operating conditions were used for the Nano Spray Dryer: nozzle size is 5.5 μm , the velocity of the gas flow is 140 $\text{L}\cdot\text{min}^{-1}$, the relative intensity of the spray is 50%, $T=70^\circ\text{C}$, $P=29\text{ hPa}$ to 30 hPa. The process was run for 25 h, and the separated powder was then calcined at 1200°C for 3 h. For comparison, the same powder was also separated using filtration through filter paper (grade F from BIK, Russia), and then dried at room temperature for 24 h and at 1200°C for an additional 5 h.

2.3 Synthesis of polymer gels

Polymer gels were prepared using the method of free-radical polymerization. These polymer gels (PG) were used in experimental studies at a suitable ratio of MMA:MAA:PEG of 29:14:57 (wt%). All reagents were placed in a cylindrical polyethylene mold ($d = 1.2\text{ cm}$, $h = 5\text{ cm}$), and the polymerization process was carried out at a temperature of 70°C for 24 h. Benzoyl peroxide, serving as the free-radical initiator, was added at a concentration of 0.05, wt% [22].

In some cases, an additional step was performed for polymer gels containing aluminum oxide powder. Before the polymerization reaction began, aluminum oxide powder was added to the monomer solution in a glass polyethylene container and stirred magnetically at 40°C for 30 min. After this pre-treatment, the same process as for the pure gel was followed. The concentration of aluminum oxide particles in the gel was varied from 0.5 wt% to 1.2 wt%.

For experiments, films of polymer gel with a thickness of 0.5 mm were prepared in two different ways. The first way is to produce films by hot pressing, for which the polymer gel is placed between the fluoroplastic plates, pressed together in clamps, and exposed to 75°C for 1.5 h. The second way is to obtain a film from a polymer gel solution in a mixed solvent containing (vol%) toluene (10), butyl acetate (15), and methyl cellosolve (75) [22].

2.4 Preparation of researched objects

Light crown (FK), heavy phosphate crown (PSK), and flint (F) were used as research objects. A solution of indene-coumarone resin in toluene and nitrocellulose enamel was used as a contaminant. 250 μL of the contaminant solution was uniformly applied to an optical glass surface ($S = 1\text{ cm}^2$) using a brush, and left to evaporate for five days, ensuring good adhesion to the surface.

2.5 Resistance measurements

The resistance measurements of the polymer matrix were taken using the Measuring instrument L, C, R universal E7-11 device (Russia). A 0.5 mm thick polymer gel sample was placed between two 1 cm^2 copper electrodes and fixed in a cell with a constant clamping force, with an alternating current of 1000 Hz.

2.6 Solvents capture

Solvent uptake studies were applied to investigate the behavior of polymer gels in various solvents, based on gravimetric measurements. The polymeric samples ($m = 50\text{ mg}$) were placed in sealed vessels containing methyl cellosolve or toluene at room temperature. At specific time intervals, the samples were removed, weighed, and then re-immersed in the solvent. The mass changes of the samples were calculated as

$$\alpha = \frac{m-m_0}{m_0} \times 100\% \quad (1)$$

where m_0 and m – the mass of the polymeric samples before and after the solvent capture.

2.7 ATR-FTIR measurements

The attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) measurements were carried out to investigate structural changes in polymer gels using a Tensor 27 spectrometer (Bruker Optik GmbH, Germany), equipped with a GaSe prism (Pike Technologies, USA).

The ATR-FTIR spectra between 4000 cm^{-1} and 500 cm^{-1} were collected at a resolution of 4 cm^{-1} by 128 scans. The data were processed in the Performance Guard program.

2.8 Microscopy measurements

Scanning electron microscope (SEM) (JEOL JSM-7500FA, Japan) with a gun power of 15.2 kV was used to estimate morphology of Al_2O_3 particles.

Optical microscope (Metam PB-21-1) and atomic force microscope (AFM) Ntegra Aura (NT MDT, Russia) were used to observe the changes on the optical glass surfaces.

AFM measurements were performed on air without additional sample preparation. An NSG01 cantilever with an average stiffness of $5.1\text{ N}\cdot\text{m}^{-1}$ and a typical resonant frequency of 150 kHz was used in the experiment. The obtained images were processed using the Gwyddion program.

2.9 The cleaning procedure by polymer gels

First method: The polymer gel film was obtained by hot pressing and was stuck on the contaminated glass surface for 1 hour at 25°C. It was then removed using tweezers (Figure 1(a)).

Second method: A polymer gel solution was poured onto the contaminated glass surface and left to dry for 2 h at 25°C until the solvent had evaporated (Figure 1(b)).

3. Results and discussion

3.1 The characterization of polymer gels

In the first part of the study, polymer gels were prepared with different concentrations of aluminum particles. Their ATR-FTIR spectra and solubility in various solvents were investigated. This allowed us to establish the approximate structure of the polymer gels containing aluminum oxide particles.

The special morphology of the Al_2O_3 nanoparticles prepared by the nano-spray drying method (Figure 2(a)) contributes to their uniform distribution in the polymer gel. On one hand, the Al_2O_3 nanocrystallites are tightly bonded in a spherical particle, on the other hand, there is almost no aggregation of particles. In contrast, the particles of the same composition obtained by precipitation and filtration (Figure 2(b)) possess substantially different properties. They are aggregated and not uniformly distributed in the polymer gel. The optical images demonstrate the difference between the surfaces of the two types of polymer gels (Figure 2(a-b)).

Figure 3 shows the resistivity measurements of the polymer gel with aluminum oxide nanopowder. As the concentration of aluminum oxide rises, the electrical conductivity of the polymer also increases. This is likely due to the fact that some of the polyethylene glycol molecules are used to solvate the aluminum particles in the polymer matrix. As a result, the carbonyl groups from MMA and MAA are released and some of the crosslinking points in the gel are reduced, making the polymer matrix more flexible. This fact provides increase the conductivity of the polymer gel. When the concentration of Al_2O_3 exceeds 1 wt%, the resistivity of the gel does not significantly change. This effect can be explained by the beginning of aggregation and sedimentation of aluminum oxide particles during the polymerization process.

To confirm the changes in the structure of the polymer gel resulting from the addition of Al_2O_3 nanoparticles, their behavior was studied in solvents of different polarities (toluene and methyl cellosolve) in terms of dissolution/swelling. Figure 3 shows the difference in rates of mass loss of polymer gels in these solvents. Two processes occur simultaneously: the release of PEG molecules from the polymer matrix and the penetration of solvent molecules into the gel.

The interaction between the polymer gel and the solvent molecules causes some hydrogen bonds within the polymer gel to break, as a result, the polymer swells. Some remaining hydrogen bonds act as additional crosslinking points in the polymer network, limiting the extent of gel swelling [18].

Polymer gels of various compositions are dissolved in MC solvent. There is a slight increase, followed by a decrease in the weight of

the polymer gel in toluene solvent. The main reason for this is that MC has a high dielectric constant and, therefore, a high polarity (the dielectric constants of MC are 15.9 and Tol is 2.3).

According to Figure 4 (curves 2, 4), the duration of the swelling and dissolution stages require less time due to the addition of aluminum oxide particles into the polymer gel. This fact confirms the assumption made above that the presence of Al_2O_3 contributes to the breaking of intermolecular bonds inside the polymer gel. Therefore, the entrance of solvent molecules into the polymer matrix and the exit of PEG from it occur rapidly enough.

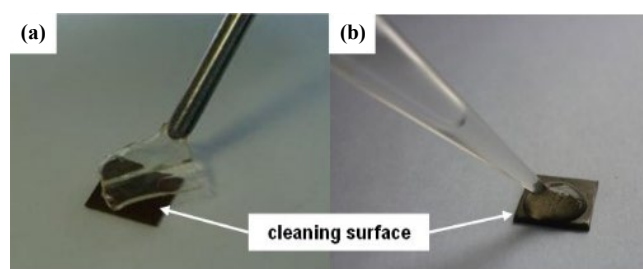


Figure 1. The cleaning procedure of a surface by the polymer gel film (a), and polymer gel solution (b).

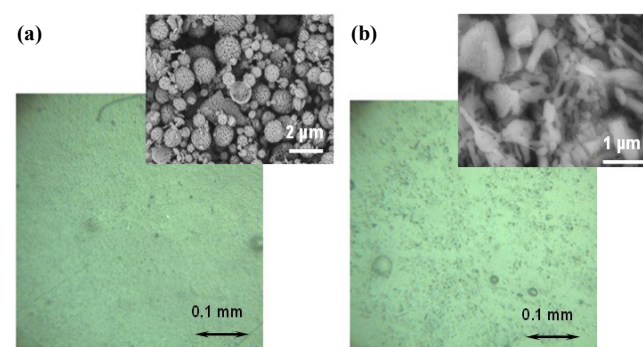


Figure 2. Optical surfaces of PG with Al_2O_3 particles (0.5 wt%). SEM images of the Al_2O_3 particles, synthesized using the nano-spray and filtration methods, are shown in images (a) and (b), respectively.

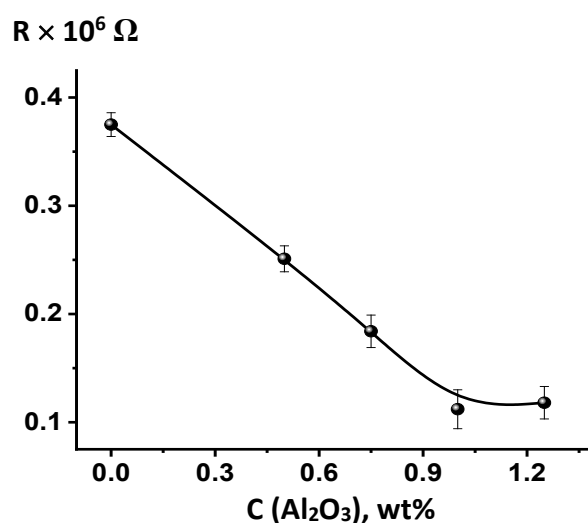


Figure 3. Electrical resistance of the polymer gel as a function of Al_2O_3 concentration (polymer film thickness is 0.5 mm).

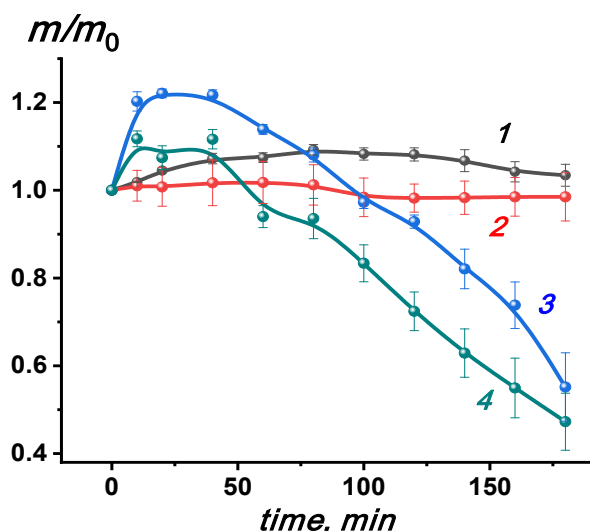


Figure 4. Change of the gel mass loss ratio into methyl cellosolve (1, 2) and toluene (3, 4): 1, 3 – PG, 2, 4 – PG- Al_2O_3 .

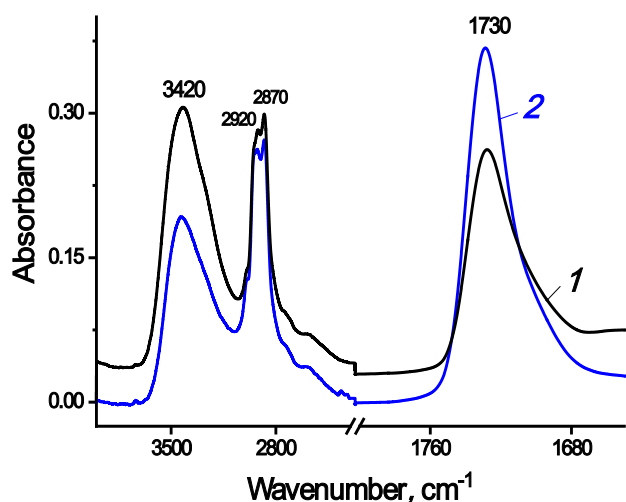


Figure 5. ATR-FTIR spectra of polymer gels: 1 – PG, 2 – PG- Al_2O_3 .

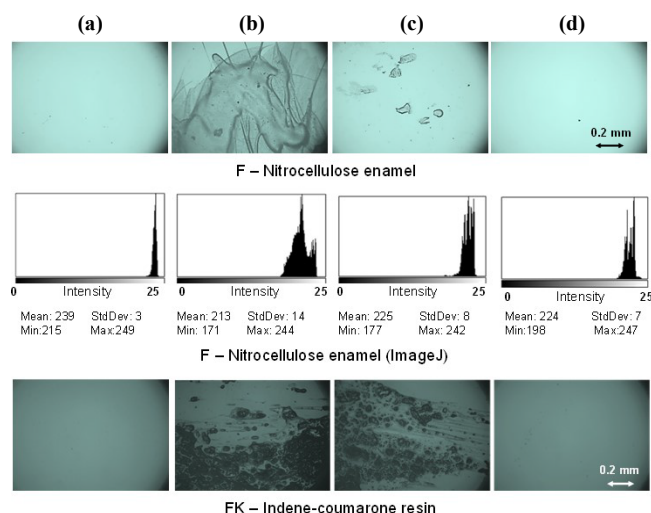


Figure 6. Images of optical glass surfaces before (a) and after (b) application of the contaminants, after cleaning with the polymer gel film (c) and polymer gel solution (d).

The structural changes in the polymer gel were assessed using ATR-FTIR spectroscopy (Figure 5). The structure of the polymer gel constitutes a network of PMMA macromolecules and partially ionized PMAA macromolecules loaded with PEG molecules. At the same time, PMMA and PMAA macromolecules can form loose tangles and linear fragments stabilized by hydrogen bonds in the matrix. PEG molecules play the role of «binder bridges» between PMMA and PMAA macromolecules, resulting in the formation of a physical network in the polymer gel. The intensity of the –OH stretching vibrations of PEG decreases, while the intensity of the –C=O stretching vibrations of MMA-MAA increases with the addition of Al_2O_3 particles to the gel. This is caused by the number of intermolecular hydrogen bonds decrease, as a consequence, the mobility of polymer chains increases. Thus, infrared spectroscopy data confirm conclusions obtained by mass loss and resistance measurement methods. These polymer gels should better clean the optical glass surfaces and easily peel-off from them because of reinforcing effect of aluminum oxide particles. Therefore, the energy difference between peeled-off gels with and without particles can be the driving force behind the peeling process of the polymer film [24,25].

A polymer gel film was stuck on the optical glass surface and left for an hour. The changes in the optical glass surfaces were examined through optical microscopy, and the degree of cleaning was quantified using ImageJ software, which calculated a histogram of gray value distribution in the image (Figure 6, second line). Figure 6 compares the results for all types of glasses.

As shown in the images of F and FK, the surface cleaned with the polymer gel solution is free of contaminants (Figure 6(d)). After the solvent evaporates, the polymer gel film is easily removed from the optical glass surface, and indene-coumarone resin and nitrocellulose enamel are completely removed. The method using polymer gel films obtained by hot pressing is less effective (Figure 6(c)), and this trend continues for all types of glasses (Figure 7(c)). The exception is flints contaminated with nitrocellulose enamel, which remain in their initial state. This method is less effective in removing indene-coumarone resin from all types of optical glasses. If the contaminants are not removed from the optical glass surface after one cleaning cycle, the cleaning can be repeated up to two cycles. As shown in Figure 7, heavy phosphate crowns are less effectively cleaned than light crowns and flints. It is well known that glasses containing relatively easily soluble network-forming substances such as boron oxide and phosphorous oxide have low chemical resistance [23].

3.2 The cleaning of optical glass surfaces by polymer gels

In the second part of the study, samples of optical glass containing various contaminants were prepared. Polymer gel solutions (MMA-MAA-PEG) and polymer gel films obtained by hot pressing were used to clean samples of optical glasses (light crowns (FK), heavy phosphate crowns (PSK), and flints (F)), contaminated with a solution of indene-coumarone resin in toluene and nitrocellulose enamel (Figure 6).

The polymer gels used for cleaning should not only be tightly bonded to contaminants, but also easily removed from cleaned surfaces. The addition of Al_2O_3 particles to the gel reduces adhesion and facilitates the removal of the gel from the surface. To determine the optimal content of matrix components, we explored the influence of Al_2O_3 and PEG

concentrations on cleaning effectiveness. An increase in the concentration of Al_2O_3 from 0.5 wt% to 1.25 wt% has no effect the cleaning efficiency. However, a high concentration of Al_2O_3 leads to particle sedimentation during the polymerization process. Therefore, 0.5 wt% Al_2O_3 is a suitable concentration for polymer gels [26].

Figure 8 shows the results of cleaning glass by polymer gels with different PEG concentrations.

The cleaning with the polymer gel containing 57.2% PEG is not sufficiently effective. On the other hand, traces remain on the optical glass when using a gel with 66.7% PEG. Therefore, 62.5% PEG in the polymer gel seems to be an appropriate concentration.

To evaluate the influence of polymer gels on the morphology of optical glass surfaces during cleaning, they were placed on the cleaned objects and removed after 10 days. Based on AFM measurements show that the polymer gel does not damage the optical glass (Figure 9), as the surface roughness of the glass remains unchanged even after prolonged contact with the gel medium. It can therefore be assumed that the gel films could be used not only for cleaning but also for preserving and protecting glass surfaces during transport or storage.

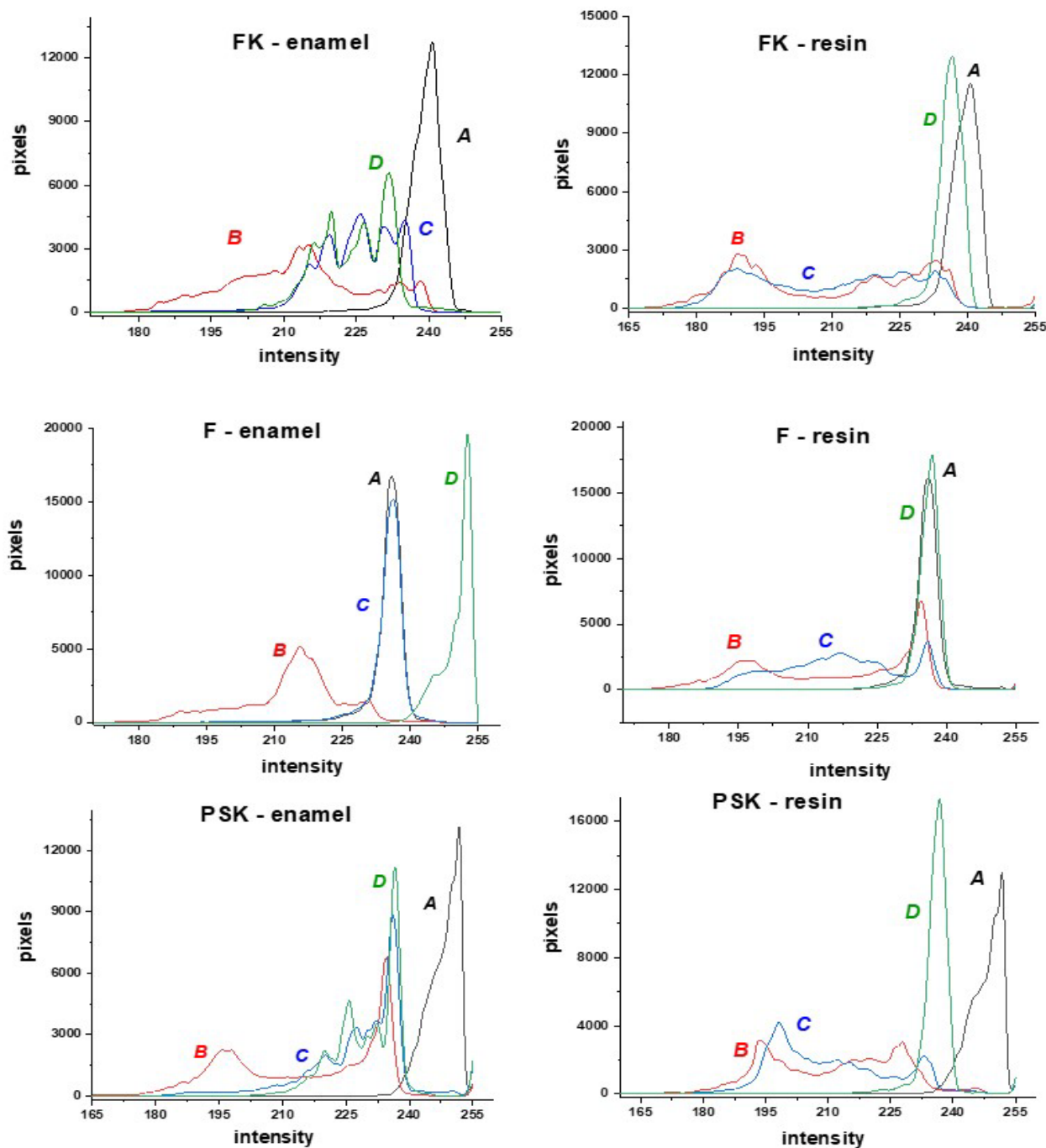


Figure 7. The histograms of the distribution of gray values on the optical glass surfaces-characterizing the cleaning before (A) and after (B) application of the contaminants, after cleaning with the polymer gel film (C) and polymer gel solution (D).

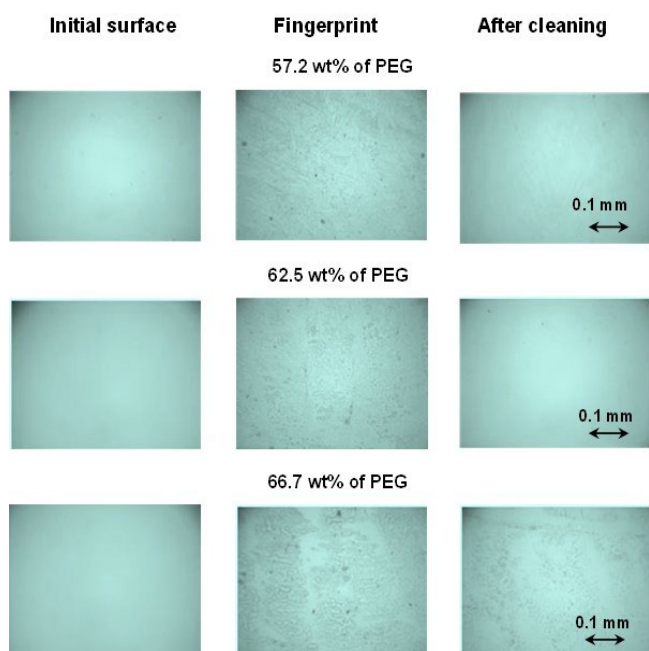


Figure 8. Images of flints surfaces after the fingerprint removal with the polymer gel film.

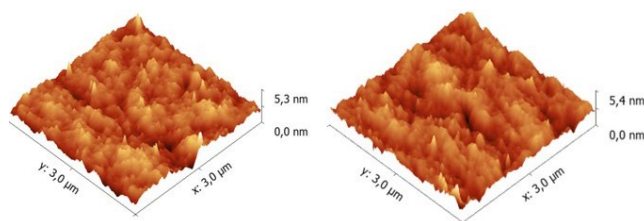


Figure 9. AFM 3D-images of optical glasses (PSK) surfaces before (a) and after (b) contact with the polymer gel in 10 days.

4. Conclusions

The method of cleaning optical glass surfaces using polymer gels based on methyl methacrylate, methacrylic acid, polyethylene glycol, and aluminum oxide particles, prepared by the nano-spray drying technique, is a promising and efficient solution. The addition of Al_2O_3 particles as a reinforcing filler to the polymer gel allows for the easier removal of the gel film from the cleaned glass surface. The optimal ratio of the MMA-MAA-PEG- Al_2O_3 gel is 25:12:62.5:0.5 (wt%).

To clean surfaces of impurities, we can use polymer gels without the need for nanoparticles. However, if it is necessary to enhance the effectiveness of the cleaning process, polymers incorporating nanoparticles can also be used. Nanoparticles can be produced not only through nano-spray drying, but also through more easily available chemical methods, ensuring the preservation of the size and morphology of the nanoparticles.

The gel demonstrates effectiveness even when very high concentrations of contaminants are used on optical glasses. The advantages of this method include its effectiveness, simplicity, use of small volumes of solvents, and ability to clean objects with any shape or surface relief. The polymer gels are prepared from easily accessible components using a simple synthesis process. The proposed cleaning method can be compared to traditional techniques using acetone as

an example, as it is one of the most effective agents for removing organic contaminants. For example, flints need to be soaked in acetone for two days at room temperature or 2 h to 3 h at 40°C to remove indene-coumarone resin from a surface. This process is less environmentally friendly and more time-consuming than the method using polymeric gel.

Credit authorship contribution statement

Galina V. Lyamina: Writing – original draft, Methodology, Writing – review and editing, Conceptualization, Supervision.

Oksana V. Dubinina: Writing – review and editing, Conceptualization, Supervision.

Alfa E. Ilela: Data curation, Investigation, Methodology, Visualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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