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TUNING THE WETTING PROPERTIES OF LOW-DENSITY POLYETHYLENE FILMS VIA CHEMICAL OXIDATION AND STEARIC ACID MODIFICATION

Impregnated surfaces have recently become a promising class of materials that overcome the limitations of classic superhydrophobic coatings and demonstrate high stability and multifunctionality in such areas of application as anti-icing, water-repellent coatings, and barrier coatings. The problem of controlling the wettability of polyethylene as a typical polyolefin substrate is considered with the aim of developing a reproducible and environmentally compatible method for regulating its surface energy. A combined strategy was applied, which included the fabrication of a template using a femtosecond laser, thermo-pressing of polyethylene to obtain textured replicas, followed by chemical oxidation with nitric acid and potassium permanganate, and final hydrophobization with stearic acid as a biologically derived alternative to fluorinated modifiers. Surface morphology, oxidation effects, and wetting behavior were investigated using microscopy and contact angle measurements. It was shown that pure polyethylene has a water contact angle of $\sim 91^\circ$, which corresponds to its nonpolar and hydrophobic nature. Oxidation significantly reduced the contact angle to 28° due to surface polarization and possible deposition of manganese oxides, while further treatment with stearic acid restored and even exceeded the initial values, yielding $\sim 96^\circ$ on flat surfaces. The results showed that texturing increased the contact angle to 135° , and in combination with oxidation and hydrophobization to 145° , which is 54° higher than the untreated material. It was shown that the modified surfaces were resistant to wetting by liquids with low surface tension, with a critical surface energy of 55 mJ/m^2 , which is considered a high threshold for non-wettability. It has been established that the key mechanism of stable hydrophobization is the fixation of stearic acid molecules by polar groups introduced during oxidation, with the external orientation of hydrocarbon tails. The approach has been found to be reproducible and does not compromise the mechanical integrity of polyethylene. Prospects for further improvement include the introduction of metal nanoparticles to form secondary textures and their subsequent hydrophobization, which may contribute to the development of superhydrophobic and infused surfaces. The results highlight a scalable methodology to produce non-fluorinated, durable, and superhydrophobic polymer surfaces.

Key words: polymer, polyethylene, wetting, chemical treatment, surface modification, heat pressing, contact angle.

Formulation of the problem. Infused surfaces represent a novel class of materials that, on one hand, overcome the inherent limitations of the previous generation of materials – namely, water-repellent textured superhydrophobic surfaces in dynamic and

mechanical stability – and, on the other hand, provide highly effective performance in applications such as ice accretion mitigation, suppression of wetting by various liquids, and protection against liquid penetration into the underlying texture. This technology is

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based on surfaces with a highly developed texture. Such substrates may be intrinsically porous, composed of interconnected fibers, or they may be artificially textured using various techniques, such as chemical etching or laser ablation. A critical requirement is that the structural pores must be capable of retaining the infused liquid within the texture [1].

The infused liquid is a crucial component of these materials, as it resides within the cavities formed by the elements of the surface structure and directly interfaces with the surrounding environment. When a droplet of a wetting liquid contacts such a surface, it essentially rests on the layer of the infused liquid rather than on the solid substrate. Consequently, during droplet removal, a portion of the infused liquid may also be displaced. Moreover, the polarity of the infused liquid significantly affects the spreading behavior of the wetting liquid on the surface and governs the specific amount of infused liquid retained within the pores, thereby determining the operational lifetime of the infused surface.

Consideration of the working mechanism of infused surfaces highlights the critical role of the affinity relationships between the porous substrate (serving as the scaffold for the infused liquid) and both the infused and wetting liquids. In scenarios where the infused liquid exhibits strong affinity to the substrate, while the wetting liquid does not, the system effectively repels the wetting liquid. In contrast, if either the infused liquid or the porous substrate shows substantial affinity toward the wetting liquid, wetting inevitably occurs. In the former case, the infused liquid may be gradually displaced and removed from the surface, whereas in the latter case, the wetting liquid can penetrate beneath the infused layer, leading to the breakdown of the system [2].

This fact underscores the relevance of investigating strategies to enhance the affinity between the infused liquid and the porous substrate in which it resides. The primary parameter that governs this affinity is the polarity or nonpolarity of both the liquid and the surface. In particular, the components of the surface tension of the infused liquid can be compared with the corresponding components of the surface energy of the solid substrate that constitutes the structural framework of the system.

For all substances, both surface energy and surface tension are determined by their chemical composition. However, this is not always the only parameter that must be considered in engineering design. For infused liquids, factors such as chemical composition, molecular weight, and the presence of impurities critically influence several technological aspects. First,

they determine the viscosity, which controls both the rate of liquid loss from the surface and the rate of self-replenishment of the infused layer. Second, the chemical composition governs the stability of the infused liquid under environmental exposure. Moreover, the chemical structure also defines the characteristic operational temperature range of the infused liquid, including its freezing point – a parameter of particular importance for anti-icing applications.

The most practical approach to controlling the affinity between the infused liquid and the substrate is to adjust the surface energy of the substrate itself. This can be achieved using a wide range of techniques and must be tailored individually for each specific type of substrate. Polymeric substrates – most notably polyethylene, which is among the most technologically convenient and economically viable materials for large-scale manufacturing – require specific approaches for tailoring their surface energy through surface modification [3].

The central problem of this study lies in controlling the wettability of polyethylene-based substrates as most promising and scalable materials for the base layer of infusion surfaces and elucidating the influence of surface texturing on this property.

Analysis of recent research and publications.

The fundamental concept of infused surfaces was first articulated in the pioneering works of Joanna Aizenberg, which were inspired by studies of natural organisms [4]. Carnivorous plants such as the pitcher plant served as a natural model, exemplifying the functional principle of infused surfaces. It is noteworthy that both in these pioneering studies and in most subsequent works, the infused surfaces and the infused liquids are typically composed of materials with inherently low surface energy and, consequently, low surface tension. In contrast, the liquids coming into contact with these surfaces usually exhibit comparatively high surface tension. One of the most common liquids that come into contact with infused surfaces is water or aqueous solutions. Water may be present in its normal liquid state, in a supercooled state, or occasionally as water vapor. However, in all reported cases of successful infused surface performance, a key factor remains the distinct difference in polarity between the infused liquid and the contacting (wetting) liquid. More practical types of infused liquids are those that combine sufficiently low surface tension with high stability under environmental stresses. For instance, when comparing aliphatic hydrocarbons with silicone-based liquids, the latter are generally more resistant to degradation by ultraviolet radiation or oxidative processes in the

presence of ozone, making them more suitable candidates for use as infused liquids. It is well known that the surface energy of polyolefins, in particular – polyethylene, is not the lowest among polymeric materials that could potentially be used as substrates. To further reduce their surface energy, a sequential strategy, that was reported in [5] for the polycarbonate substrate, was employed, beginning with controlled surface oxidation. In this approach, only the outermost layers of the material are subjected to oxidation, while the deeper layers – responsible for the bulk mechanical strength and appearance – remain unaffected. This oxidized surface layer becomes enriched with polar functional groups formed during the oxidation process. These groups are chemically reactive, which enables their use as anchoring sites for subsequent surface modifications, including hydrophobization treatments. One of the most widely used strategies for hydrophobizing oxidized polymer surfaces involves silanization. In this approach, organosilane molecules react with the polar functional groups introduced during the oxidation stage, forming covalent bonds and producing a thin siloxane-based layer on the surface. The organic moieties of the silanes can be tailored to provide hydrophobic or even low-surface-energy functionalities, thereby significantly reducing the overall surface energy of the substrate. Silanization is particularly attractive because it yields durable, conformal coatings and can be applied using relatively simple solution- or vapor-phase processes [6].

Another effective approach involves the deposition of fluorinated compounds or fluoropolymers, which impart extremely low surface energy due to the high electronegativity and low polarizability of fluorine atoms. Such coatings can be applied through various techniques, including plasma-enhanced deposition, dip-coating, or spray-coating of fluorinated precursors. Fluorinated layers provide not only enhanced hydrophobicity but also increased chemical and environmental resistance, making them suitable for long-term operation of infused surface systems.

Fluorinated organic compounds, although highly effective in reducing surface energy, are widely recognized as environmentally problematic and are often considered insufficiently sustainable. Therefore, replacing them with more environmentally compatible alternatives – even if slightly less efficient – represents a highly relevant research objective. In the present work, stearic acid was explored as a candidate for such replacement, serving as a hydrophobizing agent after the oxidation of polyethylene surfaces [7].

Increasing the surface polarity of polyethylene is a relatively straightforward and well-established task,

commonly employed, for example, in the production of printable packaging materials. In such cases, the enhancement of polarity is typically achieved using methods such as corona discharge or plasma treatment. However, the subsequent reverse hydrophobization of these surfaces, as well as the efficiency of such treatments, has been investigated only rarely. This represents an evident gap of knowledge that the present study aims to address.

Task statement. The aim of this work is to establish the possibility of flexibly controlling the wettability of textured polyolefin-based surfaces. To achieve this objective, the study addresses, in sequence, the tasks of surface texturing of polyethylene by thermopressing, enhancement of surface energy through oxidation, and subsequent modification with stearic acid to induce hydrophobization.

Outline of the main material of the study. The methodological approach of this study involved the fabrication of textured polyethylene surfaces, their subsequent oxidative treatment, hydrophobization in a stearic acid solution, and evaluation of their wettability.

To produce imprinted textures on polyethylene surfaces, a two-step procedure was employed. First, metallic templates were fabricated by creating periodic groove-like patterns on AISI304 stainless steel substrates using femtosecond laser ablation. These textured surfaces, as well as the regularities of their formation, were investigated and reported in our previous work [8].

To obtain negative replicas of the laser-textured surfaces, a solution-based method was used. 10 wt. % polycarbonate (Calibre 303 10 TNT, Dow Plastics) solutions in dichloromethane were prepared and cast onto the laser-textured steel templates, followed by solvent evaporation to form uniform thin films. These films were then mechanically peeled off from the metallic templates, resulting in negative replicas of the laser-fabricated microstructures.

Because the melting temperature of polycarbonate is significantly higher than that of LDPE, it can be used as a negative template for producing positive surface structures on polyethylene. This approach was employed in the present work: thermopressing was carried out at 120 °C, at which polyethylene melts and becomes sufficiently pliable to replicate the template features. The resulting polyethylene textures were subsequently used as substrates for the following stages of the study.

Chemical oxidation was performed using a mixture of potassium permanganate and nitric acid [9]. Solutions of each reagent were prepared at concentrations of 0.2 mol/l and volumes of 50 ml. Both textured and

non-textured polyethylene samples were immersed in this mixture overnight (approx. 10 hours). After treatment, their wettability was assessed using an optical microscope equipped with a goniometric stage.

The oxidized polyethylene samples were then subjected to hydrophobization in an isopropanol solution of stearic acid (1 wt. %) at 60 °C. The films were kept in the solution for two hours, then removed, cooled, rinsed with isopropanol to eliminate excess stearic acid, and finally dried. After drying, their wettability was evaluated.

The contact angles were measured using a Konus Academy optical microscope equipped with a goniometric stage and an UCMOS 1300 digital camera (Sigeta Optics). The measurements were performed in the TouView software, which enabled determination of the contact angle between the sample surface and the liquid droplet.

As shown in Fig. 1, the initial contact angle on the untreated polyethylene samples was relatively high, averaging around 91°. This value is consistent with previously reported literature data [10, 11] and confirms the well-established notion that polyethylene, and polyolefins in general, are inherently nonpolar and hydrophobic polymers.

Oxidation in the presence of strong oxidizing agents such as nitric acid and potassium permanganate led to a pronounced decrease in the contact angle on polyethylene surfaces, down to approximately 21°. This result primarily indicates the occurrence of direct oxidation of the polymer itself. However, it may also be partially attributed to the deposition of manganese oxide residues on the surface, which further enhances hydrophilicity, as metal oxides are generally known to exhibit intrinsically hydrophilic properties.

The surface layer, regardless of its specific composition, demonstrated moderate resistance to abrasion under normal conditions; however, under high mechanical loads it was removed together with the uppermost layer of polyethylene, thereby exposing the underlying hydrophobic material. It should be noted that this observation is of limited practical relevance for textured surfaces, as their structural features are inherently susceptible to mechanical damage.

Hydrophobization of the oxidized polyethylene surfaces in an isopropanol solution of stearic acid effectively restored their wettability to the initial levels characteristic of untreated polyethylene, with contact angles in the range of 94–96°.

Surface textures obtained by femtosecond laser ablation Fig. 2 (a, b) are anisotropic groove-asperity patterns with identical periods of 60 µm but different groove/asperity width ratio. For the texture 1 (Fig 2.a) the asperity and groove-bottom widths are about 15 µm. For the texture 3 (Fig 2.b) the asperity width is 45 µm. Their cross-sections exhibit truncated pyramid geometry. As it can be seen from Fig 2.c and Fig 2.d – the negative textures on a polycarbonate surface remain the geometrical configuration of the main patterns including period and width of its structural elements. The positive textures obtained on the surface of LDPE (Fig 2.e, f) are identical to that of the initial metal templates. The texture depth is decreased to 18 µm from 22 µm of original texture, that may be explained by increased viscosity of the polyethylene melt and not complete fulfillment of the texture void volumes. However, this depth deviation is within the error margin of measurement and is discussable.

Table 1

Static water contact angles of LDPE surfaces

Material	Water contact angle, °		
	Pristine	Oxidized	Hydrophobized
Flat	91±1,5	28±2,0	95±1,5
Texture 1 (parallel)	125±1,5	103±2	130±2
Texture 1 (perpendicular)	130±1,5	107±2	131±2
Texture 2 (parallel)	130±1,5	126±1,5	133±1,5
Texture 2 (perpendicular)	136±1,5	130±1,5	145±1,5

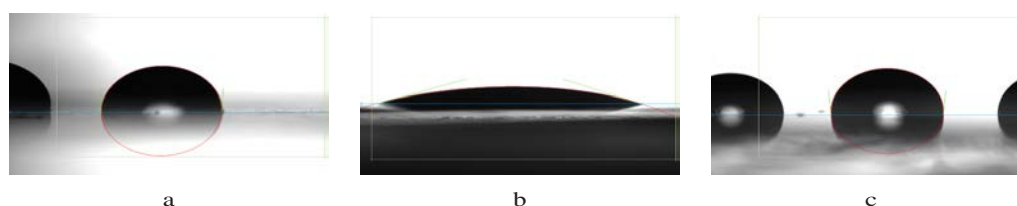


Fig. 1. Water droplet photo on the polyethylene surface: before oxidation (a), after oxidation (b), after treatment with stearic acid (c)

At the next step of the study, obtained textured surfaces of LDPE were subsequently oxidized and hydrophobized. The change of the surface energy was monitored via water contact angle measurement (Table 1). Since the obtained textures were anisotropic, the contact angle measurement may be made parallel or perpendicular to the direction of structure.

The textured polyethylene samples exhibited significantly higher contact angles compared to the flat, untreated polyethylene surface. This behavior can be explained by the Wenzel and Cassie–Baxter effects, which describe the enhancement of apparent contact angles as a function of surface roughness. At the same time, Texture #1 was less effective than Texture #2 in increasing the contact angle with water. This difference is most likely related to the structural features of Texture #1, particularly the comparatively wide spacing between its surface elements. It may be noted

that the texturing increases the water contact angle value up to 46° in comparison to a flat surface of polyethylene.

Upon oxidation, all samples demonstrated a decrease in contact angle. This effect is associated with surface polarization, namely the introduction of polar functional groups that enhance interactions with water. The trend was most pronounced for the flat polyethylene surface. In contrast, for the textured samples, the surface topography remained an effective factor in maintaining water repellency; as a result, the contact angles did not drop catastrophically but instead remained at relatively high values.

Subsequent treatment with stearic acid significantly increased the contact angles of the oxidized surfaces. The values not only returned to their original levels but in some cases exceeded them. For instance, on the flat polyethylene surface the con-

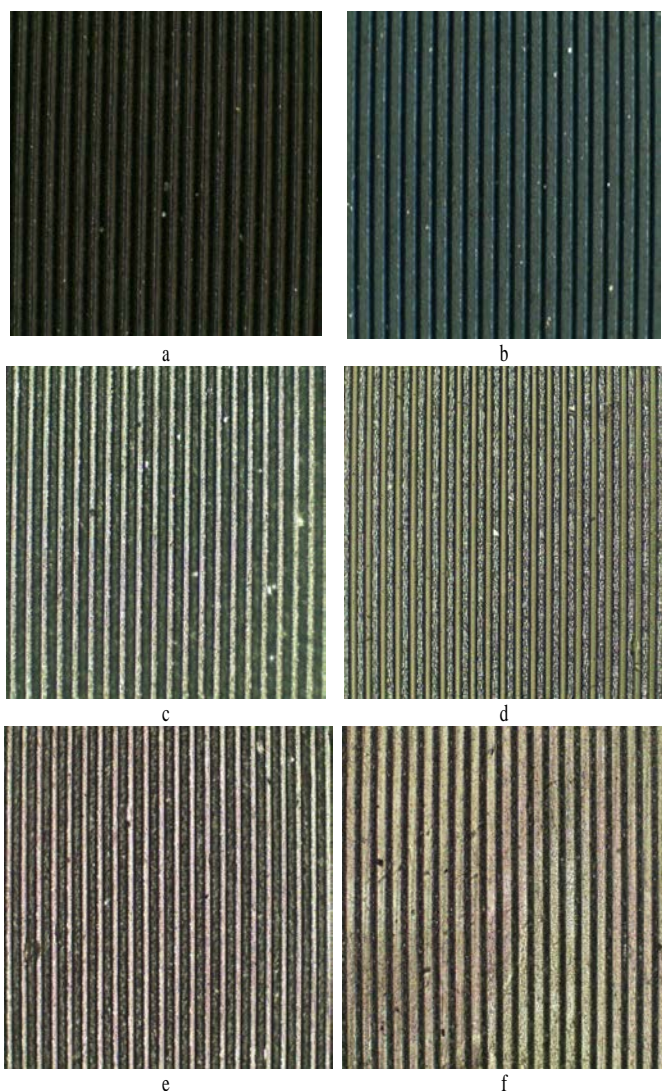


Fig. 2. Textures of samples: texture 1 (a, c, e); texture 2 (b, d, f); metal templates (a, b), polycarbonate negatives (c, d); polyethylene positives (e, f)

tact angle increased by approximately 4° compared to the untreated material, a difference beyond the experimental error and therefore statistically significant. For Texture #1, the increase ranged from 1° to 5° , depending on the measurement orientation. The most pronounced effect was observed for Texture #2, where the contact angle increased by nearly 10° in the perpendicular measurement orientation.

The obtained highest value of the water contact angle is 145° , that is 54° higher than the initial material indicating the effectiveness used texturing and treatment approach. The critical surface energy of these structures, determined as the lowest surface tension of the liquid, at which the wetting plateau remains is found to be close to 55 mJ/m^2 . This can be used as an indicator of water repellent properties stability against medium polarity liquids as well.

The contact angle data suggests that during stearic acid treatment, the molecules anchor to the oxidized polyethylene surface via polar functional groups, while the hydrophobic hydrocarbon chains orient outward. This results in the formation of a more uniform and defect-free hydrophobic layer than that of pristine polyethylene. The enhanced efficiency of the treatment is largely due to the combined effect of oxidation and subsequent hydrophobization. Oxidation with potassium permanganate may lead to the deposition of small amounts of manganese oxides (MnO_2) in the upper surface layers. These oxides can interact with stearic acid, thereby further improving the effectiveness of the surface modification.

In preliminary studies, we also attempted to hydrophobize oxidized polyethylene surfaces using alkoxysilanes. However, in this case the treatment proved to be largely ineffective, whereas stearic acid demonstrated substantially higher efficiency. An unexpected finding was that textured polymer films, when subjected to hydrophilic pretreatment, could also be functionalized with hydrophilic metal-based nanoparticles deposited onto the surface. Such modified films could subsequently be hydrophobized using agents selected for maximum compatibility with the nanoparticles, opening new possibilities for tailored surface functionalization.

Although this line of investigation requires further research and development, it represents a promising

and, importantly, reproducible approach compared to simple polarization of polyethylene surfaces by oxidation. A major advantage of this method is that the polymer itself is not degraded, allowing it to retain its mechanical properties, operational lifetime, and environmental resistance.

Conclusions. In this work, a method for regulating the surface energy of polyethylene was demonstrated through a sequence of oxidation and subsequent hydrophobization. Oxidation reduced the contact angle from 91° to 28° , while hydrophobization restored and even increased it to 96° on flat surfaces. Surface texturing alone raised the water contact angle to 135° , and when combined with the oxidation–hydrophobization cycle, the contact angle reached 145° , which is 54° higher than that of pristine polyethylene.

The modified materials were shown to be resistant to wetting by liquids with reduced surface tension. The critical surface energy at which wetting commenced was determined to be 55 mJ m^{-2} , a comparatively high threshold value. This demonstrates that the combination of texturing and chemical functionalization provides a synergistic effect, enabling the creation of highly non-wettable surfaces.

A promising direction for extending these results lies in the incorporation of nanoparticles, such as metallic nanoparticles, to generate secondary textures on polyethylene replicas fabricated by the template method. These nanoparticles can be readily functionalized with simple hydrophobizing agents, including stearic acid, which may further enhance the performance and allow the fabrication of superhydrophobic surfaces using the same principle.

Finally, porous surfaces present themselves as an excellent platform for the design of infused surfaces. As demonstrated, their polarity – and therefore their affinity toward infused liquids – can be tuned on demand, offering additional flexibility for developing advanced liquid-repellent and multifunctional materials.

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Миронюк О.В., Баклан Д.В., Савченко Д.О., Пашенко Є.О. РЕГУЛЮВАННЯ ВЛАСТИВОСТЕЙ ЗМОЧУВАННЯ ПЛІВОК З ПОЛІЕТИЛЕНУ НИЗЬКОЇ ЩІЛЬНОСТІ ЗА ДОПОМОГОЮ ХІМІЧНОГО ОКИСНЕННЯ ТА МОДИФІКАЦІЇ СТЕАРИНОВОЮ КИСЛОТОЮ

Просочені поверхні нещодавно стали перспективним класом матеріалів, які долають обмеження класичних супергідрофобних покриттів і демонструють високу стабільність та багатofункціональність у таких сферах застосування, як антиожедні, водовідштовхувальні покриття та бар'єрні покриття. Розглянуто проблему контролю змочуваності поліетилену як типового поліолефінового субстрату з метою розробки відтвореного та екологічно сумісного способу регулювання його поверхневої енергії. Застосовано комбіновану стратегію, що включала виготовлення шаблону за допомогою фемтосекундного лазера, термопресування поліетилену для отримання текстурованих реплік, подальше хімічне окислення азотною кислотою та перманганатом калію, а також остаточну гідрофобізацію за допомогою стеаринової кислоти як біологічно похідної альтернативи фторованим модифікаторам. Досліджено морфологію поверхні, ефекти окислення та поведінку змочування за допомогою мікроскопії та вимірювання кута змочування. Показано, що чистий поліетилен має кут змочування водою $\sim 91^\circ$, що відповідає його неполярній та гідрофобній природі. Окислення значно зменшило кут змочування до 28° через поляризацію поверхні та можливе осадження оксидів марганцю, тоді як подальша обробка стеариновою кислотою відновила і навіть перевищила початкові значення, давши $\sim 96^\circ$ на плоских поверхнях. Результати показали, що текстурування збільшило кут змочування до 135° , а в поєднанні з окисленням гідрофобізацію до 145° , що на 54° перевищує показники необробленого матеріалу. Показано, що модифіковані поверхні були стійкими до змочування рідинами з низьким поверхневим натягом, з критичною поверхневою енергією 55 мДж/м^2 , що вважається високим порогом для незмочуваності. Встановлено, що ключовим механізмом стабільної гідрофобізації є закріплення молекул стеаринової кислоти за допомогою полярних груп, введених під час окислення, з зовнішньою орієнтацією вуглеводневих хвостів. Виявлено, що підхід є відтворюваним і не порушує механічної цілісності поліетилену. Перспективи подальшого вдосконалення включають введення металевих наночастинок для формування вторинних текстур та їх подальшу гідрофобізацію, що може сприяти розвитку супергідрофобних та інфузованих поверхонь. Результати підкреслюють масштабовану методологію виробництва нефторованих, стійких та супергідрофобних полімерних поверхонь.

Ключові слова: полімер, поліетилен, змочування, хімічна обробка, модифікація поверхні, термопресування, кут змочування.

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