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DEVELOPMENT OF CORN STALK- AND COCONUT SHELL-BASED FILLERS AND THEIR UTILIZATION IN POLYLACTIDE COMPOSITES

The transition from petroleum-based polymers to biological alternatives is one of the key strategies for reducing carbon footprint and dependence on fossil resources. A literature review has shown that although polylactide (PLA) is a commercially available and widely studied biopolymer, its use in water-based dispersion coatings remains limited due to the lack of suitable dispersion preparation methods and poor compatibility of polar cellulose fibers with the nonpolar PLA matrix. The article is devoted to the utilization of agricultural waste as fillers and shows that this approach is a sustainable way to improve PLA-based materials while solving the problem of waste disposal within the circular economy. The aim of the work was to develop aqueous PLA dispersion coatings reinforced with fibrous fillers obtained from corn stalks and coconut shells. The fillers were obtained by mechanical grinding, chemical pretreatment, and fractionation, followed by silane modification to improve hydrophobicity. Fibers with a size of 30-400 μm (average size ~65 μm for corn, ~150 μm for coconut) were obtained, which were incorporated into PLA dispersions stabilized with sodium polyacrylate and processed into films by spin coating. The morphology of the filler, surface chemistry, dispersion quality, and coating formation were evaluated using structural, spectroscopic, and microscopic methods. The results showed that silane treatment increased the water contact angle to 133° for corn fibers and 140° for coconut fibers, which corresponds to the Cassie-Baxter effect. IR spectroscopy results confirmed a decrease in the adsorbed water content. Uniform fiber distribution and complete particle fusion at 130 °C were demonstrated. Studies showed that moisture absorption decreased from 3,61 wt. % to 2,81 wt. % (\approx 30%) for corn-based composites and from 3,33 wt.% to 3,0 wt. % (\approx 10%) for coconut-based composites, indicating improved water resistance and stronger interfacial interactions in the case of corn fibers. The possibility of producing fully biobased coatings with low VOC content, reinforced with fibrous fillers obtained from agricultural waste, has been established. This approach has been shown to provide a platform for extending PLA dispersion technology to other agricultural wastes and alternative bio-based matrices, such as polyhydroxyalkanoates. Optimization is proposed, aimed at developing industrial formulations by incorporating plasticizers, pigments, stabilizers, and fully biocompatible surface modifiers to obtain scalable and durable bio-based coating systems.

Key words: cellulose, polymer, fibers, polylactide, composite, particles.

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Formulation of the problem. One of the effective approaches to substantially reducing the carbon footprint and lowering the reliance on fossil fuels is the transition from petroleum-based polymers, which currently account for most polymeric materials produced worldwide, to bio-based alternatives. Among these, polylactide (PLA) is considered one of the most promising polymers, as it is not only extensively studied but also technologically developed and commercialized. Chemically, PLA is a polyester by nature. Being a thermoplastic, it can serve as a substitute for various polyolefins and polyesters in a wide range of applications, from 3D printing to packaging materials [1].

In recent years, PLA has found increasing use in various industrial applications. Nevertheless, its utilization as coating material remains relatively limited [2]. This is primarily because PLA is commonly processed into films from solution, a method that requires the use of volatile organic solvents. Dispersion-based systems of PLA, however, face certain challenges, as this polymer cannot be synthesized via emulsion or suspension polymerization techniques.

Research efforts have been directed toward the development of aqueous dispersions of PLA, which advance this material into the category of low-VOC systems and address the limitations associated with solvent-based PLA. Several studies have investigated the preparation of PLA aqueous dispersions using the solvent evaporation method. Researchers who proposed this method primarily focused on obtaining the dispersion itself and demonstrated only the feasibility of forming films from such dispersions. However, for fully functional coating material additional functional components are required [3].

As is well known, paint and coating formulations may contain up to seven to ten different functional components [4]. Among these, fillers are of particular importance, as they not only reduce the overall cost of the coating but also impart specific functional properties, such as reinforcement and improved mechanical strength. The primary film-forming material used in such coatings is PLA. The filler is also expected to meet the requirement of being bio-based and derived from renewable resources.

Fillers derived from agricultural waste are particularly suitable, as they contain no fossil-based carbon and consist exclusively of biogenic carbon. Moreover, their utilization contributes to the integration of such waste streams into the circular economy. Plant-derived materials are complex and structurally diverse, and the isolation of cellulose fibers from them presents a considerable challenge that must

be addressed depending on the specific type of raw material used. Furthermore, the surface of cellulose fibers is inherently hydrophilic, which may pose an additional obstacle to their effective integration into PLA matrix [5].

The primary objective of this study is the extraction of biofibers from agricultural waste and the subsequent enhancement of their compatibility with PLA-based matrices, particularly aqueous-dispersion systems.

Analysis of recent research and publications. PLA-based composites have found wide application across various industrial sectors and are most processed by extrusion. The products of such processing include filaments for 3D printing, packaging films, and a variety of other materials [6].

In most cases, the materials used are filled grades of PLA. The processing temperature, depending on the specific grade, typically ranges from 150–160 °C up to about 180 °C. These composites are commonly filled, and it would be unusual if they were not reinforced with natural fillers, such as ground wood flour, which is also regarded as a renewable resource [7]. However, it cannot be classified as an agricultural waste material.

In composites containing cellulose-based fillers, researchers have long noted the problem of non-uniform distribution of such particles within the polymer matrix. This issue primarily arises from the mismatch in polarity between the surface of the cellulose fibers and the polymer itself, as the latter is significantly less polar than the cellulose filler. This challenge can be addressed by introducing compatibilizing agents capable of forming interfacial bridges between the filler surface and the PLA matrix [8]. One such material is maleic anhydride, as well as grafted polymers produced using this technology.

When transitioning from extrusion techniques to water-dispersed coatings, an additional challenge arises: the primary medium in which the filler is dispersed is water, a highly polar solvent. Consequently, nonpolar particles tend to disperse poorly in such an environment. In this field, the issue is typically addressed by incorporating specialized surfactant-type dispersing agents, which reduce the effective surface polarity of the hydrophobized filler and thereby facilitate its dispersion in the aqueous medium [9].

As can be anticipated, this situation results in a highly multicomponent system with numerous potential issues. For example, if a considerable amount of dispersant remains in the cured coating, it may act as a site that attracts additional water during service, thereby accelerating the degradation of the coating. In any case, the key criterion for the successful integration of cellulose-based fillers into polymer matrices – ranging from extrusion technology to latex coating systems – is the uniform distribution of particles within the matrix. Achieving this uniformity is the primary objective of the work presented in this study.

It should be noted that many studies focus on cellulose-based fillers derived from renewable resources, such as wood flour or cotton fibers. However, these materials are not agricultural waste. The reason lies in the fact that the primary raw materials are cleaner and more easily processed, whereas agricultural wastes are considerably more difficult to handle. Materials that require additional utilization hold a certain value, which makes this approach particularly promising. In our study, special attention is given to the use of agricultural residues such as corn stalks, which represent a widely available waste stream, including in Ukraine.

Corn stalks are characterized by a relatively regular structure of elementary cellulose formations, which makes them highly suitable candidates for use in PLA composites. Moreover, their utilization would enable the transfer of this experience into the broader global practice of producing fillers for PLA-based composites. A considerable number of studies have been devoted to the compounding of PLA, particularly for extrusion. Research has also been conducted on the preparation of PLA dispersions, as well as on the production and hydrophobization of fillers derived from corn residues and coconut waste. However, to date, there is no comprehensive study that describes the fabrication of films from aqueous dispersions incorporating bio-based fillers [10].

This opens the possibility of obtaining a new class of materials with virtually zero fossil carbon content, in which all components are bio-based, and the resulting coatings are entirely free of volatile organic compounds.

Task statement. The main objective of this study is the development of a novel material based on aqueous dispersions of PLA combined with biobased fibrous fillers obtained through the valorization of agricultural waste. The specific objectives of this study are as follows: to obtain fibrous fillers from agricultural waste, namely corn stalks and coconut shells; to modify the surface energy of these fillers in order to reduce their surface polarity; to incorporate the prepared fibrous fillers into PLA-based compositions in the aqueous phase using dispersing agents; and to investigate the uniformity of fiber distribution within the PLA matrix.

Outline of the main material of the study. To obtain the fillers, the raw materials, namely agricultural waste such as corn stalks and coconut shells, were first subjected to size reduction. The corn stalks were simply cut into flakes approximately 1 mm in length, whereas the coconut shells were initially crushed in a jaw crusher and subsequently further reduced in a hammer mill once the particle size reached several millimeters.

The corn stalk sample was subsequently treated with a mixture of acetic acid and hydrogen peroxide in a 70:30 volume ratio for two hours at 95 °C, followed by cooling. It was then immersed in a 0,1 M potassium hydroxide solution for two days at room temperature, after which it was treated with hydrogen peroxide.

The fiber processing algorithm was the same for both materials. Both pretreated fibers, after being washed to remove residues of the reaction mixture, were passed through a colloidal mill FDM-Z-150 and then through a blender. Following this disintegration treatment, they were fractionated by a sieving (mesh) method under a flow of water. Thus, the working size range of the filler fractions was selected to be between 30 and 400 μ m.

For the obtained cellulose fillers, which had been classified after milling and subsequently dried, a silane-based modifier was employed, namely polymethylhydrosiloxane (XIAMETER MHX-1107 Fluid 30 cSt, Dow Chemical). The fibers were mixed with a 1 wt. % solution of hydrophobizer in xylene and exposed for 3 hours, after which they were removed from the solution, washed, and dried at 135 °C to ensure the fixation of the siloxane on the surface of the cellulose fibers.

The PLA dispersions (based on 4060D PLA from Ingeo, USA) were synthesized according to the procedure previously described in our previous paper [11]. For blending with the fibers 0,5 wt. % sodium polyacrylate dispersant was added to the dispersion. The hydrophobized fibers were then introduced into this solution, where they became wetted and incorporated into the dispersion matrix.

The films were produced using the doctor blade method. A drop of the prepared material was applied to the surface and then spread through a gap of the desired thickness to form a uniform coating. The coating was subsequently dried at room temperature to remove moisture completely. Since the film formation temperature of PLA is relatively high (130 °C for the unmodified material) the obtained coating was further heated at 130 °C and held for a defined period to allow particle coalescence.

The quality of fiber surface treatment with silane was evaluated by measuring the water contact angle. For this purpose, the material was spread as a layer approximately 1 mm thick on a microscope glass slide. A flat surface was formed on top, onto which a droplet (5 μ l) of water was deposited. The contact angle was recorded automatically using a tensiometer (BGD190, Bigued Precise Instruments, China).

The compositions were analyzed using infrared spectroscopy in the range of 4000-400 cm⁻¹ (Specord IR75). For powders, the KBr pellet method was used, while for films, direct transmission measurements were performed.

The sintering of PLA particles, the composite uniformity, and fibers structure was examined using a Konus Academy optical microscope with UCMOS 1300 digital camera (Sigeta Optics) and ToupView software.

Fig. 1 shows that the processing of both corn stalks and coconut shells yields anisotropic, elongated fibers with the potential to serve as reinforcing agents. The corn-derived fibers are more diverse in both morphology and aspect ratio. They include not only relatively thin fibers but also cellulose plates resulting from the preparation process (Fig. 1, a). In the case of coconut, the fibers exhibit a more uniform width and a distinct aspect ratio (Fig. 1, b) with characteristic internal structure.

The fiber particle size distribution was determined by 300 particles analysis. The results indicate that the average particle size is approximately 65 µm for corn fibers and 150 μm for coconut fibers. As shown by the granulometric curves, the distributions are uniform and monomodal (Fig. 2). For corn fibers, the minimum particle size in the sample was 30 μm (Fig. 2, a), whereas for coconut fibers it was 70 μm (Fig. 2, b). Accordingly, most of the fractions fall within the range of 30–150 μm for corn fibers and 70–300 μm for coconut fibers.



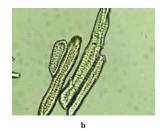


Fig. 1. Fibrous filler particles derived from: corn stalks (a); coconut shells (b)

Both materials consist predominantly of cellulose polymer, as confirmed by the presence of – OH groups (Fig. 3, a, b), with stretching absorption bands in the range of 3000–3400 cm⁻¹ and deformation vibrations near 1600 cm⁻¹. The spectra also clearly indicate the carbonaceous nature of the materials, as evidenced by C-H vibrations at 2900–2860 cm⁻¹. Hydrophobization (Fig. 3, c, d) does not significantly alter the overall spectral pattern of the materials. Only minor changes can be observed, such as the slight appearance of C-H related doublets and variations in the peak ratios associated with adsorbed water, as seen in the 1600 cm⁻¹

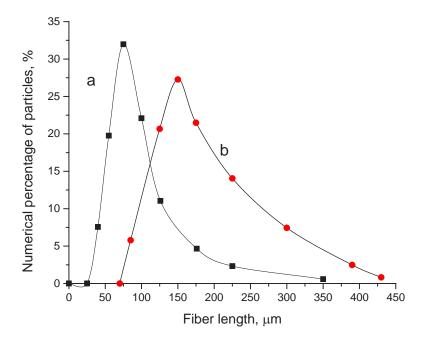


Fig. 2. Granulometry curves of particles derived from: corn stalks (a); coconut shells (b)

region indicating a reduced content of adsorbed water in the hydrophobized samples. Overall, the spectrum remains quite similar.

Hydrophobic treatment effectiveness is demonstrated on Fig. 4. The water droplet of hydrophobized corn fibers is 133° (Fig. 4, a), the contact angle of hydrophobized coconut fibers reaches 140° (Fig. 4, b). Such high contact angle values do not reflect the intrinsic wettability of the fiber surface alone. Rather, this effect arises from the porous capillary structure formed by the fibers, which enables the Cassie–Baxter state [12]. Nevertheless, these high contact angles are only attainable when the material itself possesses considerable inherent hydrophobicity (95–100°) which fully corresponds to the hydrophobicity imparted by silane treatment.

During the film formation process, the mechanism of film development from dispersed particles was first investigated. As shown in Fig. 5, a, a microscopic image of films obtained by the solvent evaporation method is presented. Fig. 5, b and Fig. 5, c illustrates intermediate stages of particle coalescence, which begin at 130 °C and progress over time. Fig. 5, b corresponds to 30 seconds of exposure at this temperature, while Fig. 5, c corresponds to 3 minutes. Fig. 5, d shows an almost fully formed film with some residual porosity after 5 minutes of thermal treatment at 130 °C. With further holding, a defect-free continuous film is obtained.

Fig. 6, a, b shows final versions of fully sintered films at 130 °C containing fibers derived from corn stalks. The fibers are distributed almost uniformly across the entire coating without forming aggregates. This indicates that the affinity between the polymer matrix and the fibers is relatively high.

The composites obtained from PLA with fibers also confirm a strong interaction between the components (Fig. 7, a, b). Within the spectral region of 3700–3500 cm⁻¹, a characteristic doublet can be observed, consisting of two sharp peaks. This feature

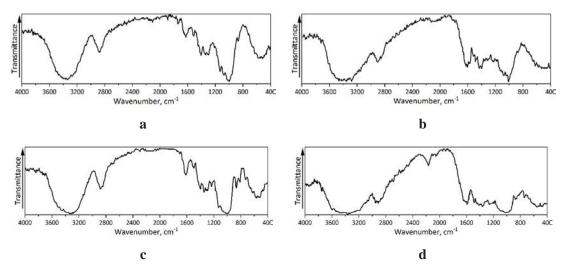


Fig. 3. Infrared spectra of fibers: pristine (a, b); hydrophobized (c, d); corn stalk (a, c); coconut shell (b, d)



Fig. 4. Water contact angle on the surface of hydrophobized pressed layer of filler: corn stalk (a); coconut shell (b)

corresponds to OH groups located chemically on the fiber surface rather than OH groups of adsorbed water. This indicates that the presence of PLA reduces the adsorption capacity of the filler, which in turn reflects a relatively high level of interfacial interaction.

This effect is particularly evident in Table 1. For the coconut-based composite containing non-hydrophobized fibers, the material sorbs 3,33 wt. % of moisture from the air. Upon hydrophobization, this value decreases by about 10%, demonstrating a measurable improvement. By contrast, the effect is much more pronounced for corn-based fillers: the non-hydrophobized material sorbs 3,61 wt. % moisture, while the hydrophobized counterpart absorbs only 2,81 wt. %. In this case, the reduction in sorption is nearly 30%, which is a substantial improvement.

Table 1 **Moisture absorption of the composites**

№	Fiber type	Fiber content, wt.	Hydrophobic treatment	Moisture uptake, wt.
1	Coconut	20	-	3,33
2	Coconut	20	+	3,05
3	Corn	20	-	3,61
4	Corn	20	+	2,81

This phenomenon can be explained by comparing the granulometry of coconut and corn fibers (Fig. 2). Corn fibers contain a significant proportion of fine particles with higher specific surface area. The larger the specific surface area, the greater the number of

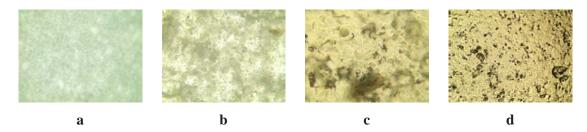


Fig. 5. Stages of PLA film sintering: pristine particles of PLA powder (a); sintering onset (30 s) (b); after 3 min (c); after 5 min of sintering (d)

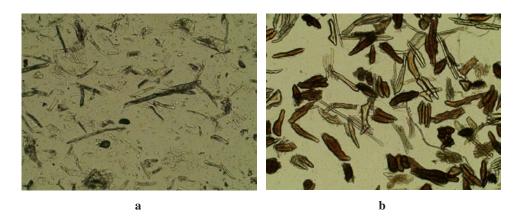


Fig. 6. Hydrophobized fiber distribution inside sintered PLA films: corn stalk (a); coconut shells (b)

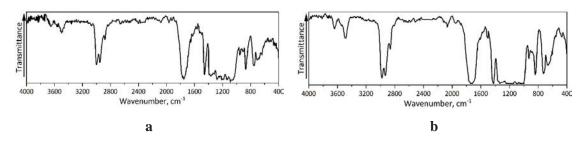


Fig. 7. Infrared spectroscopy of PLA composites with 20 wt. % filler: corn stalk (a); coconut fiber shell (b)

OH groups available for silane interaction, and consequently the higher the hydrophobization efficiency. Thus, for both coconut and corn fibers, hydrophobization proves to be an effective method for reducing moisture uptake, with particularly strong efficiency in the case of corn-based fillers.

The development of water-dispersion coatings based on PLA filled with plant-derived fillers from agricultural waste opens new prospects for utilizing a broad class of agricultural residues. These may include by-products from the processing of various fibers, such as flax or tow, as well as other agricultural fibers. Furthermore, the results of this work can be adapted to different types of fibers available in other regions, for example, banana stem residues. This approach may also prove valuable when extending the concept to alternative polymer matrices. In addition to PLA, polyhydroxyalkanoates (PHAs) may also be employed, and with the selection of an appropriate PHA, the same technological strategy could be replicated to obtain such coatings.

A promising direction for the further development of this technology lies in its refinement toward industrial-level coatings. Beyond fillers, other functional components must also be considered, such as plasticizers to reduce the glass transition temperature and facilitate film formation. Additional components could include pigments, additives to improve flow and leveling of the coating layer, as well as thickeners or dispersion stabilizers to ensure long-term stability.

Another promising avenue is to replace the surface treatment of such fillers with more bio-compatible agents, such as fatty acids or oxalates, thereby ensuring that the entire system remains fully bio-based. Moreover, this treatment could potentially be carried out in aqueous solutions rather than with organic solvents, as was done in the present work. Such modifications represent one of the prospective directions for the further advancement of this approach.

Conclusions. In this study, water-dispersion coatings based on PLA and bio-based fibrous fillers derived from agricultural waste (corn stalks and

coconut shells) were successfully obtained. The fillers were processed into fractions with particle sizes ranging from 30 to 400 μm , with average dimensions of $\sim\!65~\mu m$ for corn fibers and $\sim\!150~\mu m$ for coconut fibers. Microscopic observations confirmed their anisotropic morphology, enabling their use as reinforcing agents in PLA matrices.

Surface modification with silane significantly increased fiber hydrophobicity, as demonstrated by contact angle measurements of 133° for corn fibers and 140° for coconut fibers, attributed to the Cassie–Baxter effect. FTIR analysis indicated reduced adsorbed water content in the modified fibers and strong interfacial interactions with PLA. Composite films exhibited uniform fiber distribution and efficient particle coalescence at 130 °C, resulting in continuous coatings.

Moisture absorption tests confirmed the effectiveness of hydrophobization: for coconut fibers, sorption decreased from 3.33 wt% to ~3.0 wt% (a 10% reduction), while for corn fibers it decreased from 3.61 wt% to 2.81 wt% (a 30% reduction). These results demonstrate that corn-based fillers, due to their finer granulometry and higher specific surface area, exhibit particularly strong improvements in moisture resistance.

The findings highlight the potential of converting agricultural residues into high-value functional fillers for bio-based PLA coatings. Future research should focus on extending this approach to other agricultural fibers (e.g., flax, banana stems), employing alternative bio-based matrices such as PHAs, and replacing silane modifiers with fully biocompatible agents like fatty acids or oxalates. Further development should also target industrial-level formulations through the inclusion of plasticizers, pigments, stabilizers, and other functional additives to achieve durable and scalable coating systems.

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Миронюк О.В., Наумчик А.О., Баклан Д.В., Лінючева О.В., Сікорський О.О. РОЗРОБКА НАПОВНЮВАЧІВ НА ОСНОВІ КУКУРУДЗЯНИХ СТЕБЕЛ І КОКОСОВОЇ ШКАРАЛУПИ ТА ЇХ ВИКОРИСТАННЯ У ПОЛІЛАКТИДНИХ КОМПОЗИТАХ

Перехід від полімерів на основі нафти до біологічних альтернатив є однією з ключових стратегій для зменшення вуглецевого сліду та залежності від викопних ресурсів. Літературний огляд показав, що полілактид (PLA) хоч і є комерційно доступним та широко дослідженим біополімером, але його застосування у водних дисперсійних покриттях залишається обмеженим через відсутність відповідних методів підготовки дисперсії та погану сумісність полярних целюлозних волокон з неполярною матрицею PLA. Стаття присвячена утилізації сільськогосподарських відходів як наповнювачів і показано, що даний підхід є стійким способом поліпшення матеріалів на основі PLA, одночасно вирішуючи проблему утилізації відходів в рамках циркулярної економіки. Метою роботи було розроблення водних дисперсійних покриттів PLA, армованих волокнистими наповнювачами, отриманими з кукурудзяних стебел і кокосової шкаралупи. Наповнювачі були отримані шляхом механічного подрібнення, хімічної попередньої обробки та фракціонування, з подальшою модифікацією силаном для поліпшення гідрофобності. Отримано волокна розміром 30-400 мкм (середній розмір~65 мкм для кукурудзи,~150 мкм для кокоса), які було включено в дисперсії PLA, стабілізовані поліакрилатом натрію, і перероблені в плівки методом ракельного розпилення. Оцінено морфологію наповнювача, хімію поверхні, якість дисперсії та формування покриття за допомогою структурних, спектроскопічних та мікроскопічних методів. Результати показали, що обробка силаном збільшила кут змочування водою до 133° для кукурудзяних волокон і 140° для кокосових волокон, що відповідає ефекту Кассі-Бакстера. Результати ІЧ спектроскопії підтвердили зменшення вмісту адсорбованої води. Продемонстровано рівномірний розподіл волокон і повне злиття частинок при 130 °С. Дослідження показали, що поглинання вологи зменшилося з 3,61 мас. % до 2,81 мас. % (\approx 30%) для композитів на основі кукурудзи та з 3,33 мас. %до 3,0 мас. % (\approx 10%) для композитів на основі кокоса, що свідчить про поліпшену водостійкість та сильніші міжфазні взаємодії у випадку кукурудзяних волокон. З'ясовано можливість виробництва повністю біооснованих покриттів з низьким вмістом ЛОС, армованих волокнистими наповнювачами, отриманими з сільськогосподарських відходів. Показано, що цей підхід забезпечує платформу для поширення технології диспергування РLА на інші сільськогосподарські відходи та альтернативні біоосновані матриці, такі як полігідроксиалканоати. Запропоновано оптимізацію, що спрямована на розробку промислових рецептур шляхом включення пластифікаторів, пігментів, стабілізаторів та повністю біосумісних модифікаторів поверхні для отримання масштабованих і довговічних біооснованих систем покриттів.

Ключові слова: целюлоза, полімер, волокна, полілактид, композит, частинки.

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