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FILLERS ON THE SILICA BASE FOR POLYMER COMPOSITES FOR CONSTRUCTIONAL PURPOSES

Oleh S. Kabat[1,2]*, Krystyna V. Heti[3], Ihor L. Kovalenko[1], Anatoliy M. Dudka[1]

[1]Ukrainian State University of Chemical Technology, Gagarina Avenue, 8, Dnipro, 49005, Ukraine
[2]Dnipropetrovsk State Agrarian and Economic University, Iefremova Avenue, 25, Dnipro, 49600, Ukraine
[3]Oles Honchar Dnipro National University, Gagarina Avenue, 72, Dnipro, 49005, Ukraine

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Abstract

The finding a filler for the creation of polymer composite (PC) for constructional purposes, which is able not only for improving the level of polymer matrix properties, but also significantly for reducing its price was conducted. It was determined that such fillers are silicas of various modifications, which due to the developed surface and active "silanol" groups on it are capable of physical and chemical interaction with the polymer matrix. The most common industrial brands of silicas, such as aerosil, white soot and silica gel were selected for research. As a result of microscopic investigations it was obtained, that particles of silica of such brands as aerosil and white soot, both in the initial form and in the polymer matrix are actively aggregated, and create agglomerates up to tens of microns. This phenomenon is not observed when using silica gel. Therefore, it is more appropriate to be used as the fillers for PC for construction purposes than other investigated materials. The method of synthesis of silica gel with a high degree of chemical purity was presented. Comparative investigations of the physico-mechanical, thermophysical and tribotechnical properties of constructural polymer composites, based on aromatic polyamide, filled by industrial and synthesized silica gel were performed. It was obtained that PCs with synthesized silica gel have higher level of properties than industrial ones. This is due to the fact that the primes presented in industrial silica gel reduce its reinforcing effect on the polymer matrix and adversely affect the friction process during the friction interaction of the obtained PC with steel.

Keywords: silica; silica gel; polymer; aromatic polyamide; polymer composites; physico-mechanical properties.

НАПОВНЮВАЧІ НА ОСНОВІ ДІОКСИДУ КРЕМНІЮ ДЛЯ ПОЛІМЕРНИХ КОМПОЗИЦІЙНИХ МАТЕРІАЛІВ КОНСТРУКЦІЙНОГО ПРИЗНАЧЕННЯ

Олег С. Кабат[1,2], Кристина В. Геті[3], Ігор Л. Коваленко[1], Анатолій М. Дудка[1]

[1]ДВНЗ Український Державний Хіміко-Технологічний Університет, просп. Гагаріна, 8, м. Дніпро, 49005, Україна
[2]Дніпровськький державний аграрно-економічний університет, вул. С. Єфремова, 25, м. Дніпро, 49600, Україна
[3]Дніпровський національний університет імені Олеся Гончара, просп. Гагаріна, 8, м. Дніпро, 49005, Україна

Анотація

Проведено аналіз щодо визначення наповнювача для створення полімерних композиційних матеріалів (ПКМ) конструкційного призначення, який здатний не тільки покращити рівень властивостей полімерної матриці, а і значно здешевіти її. Визначено, що такими наповнювачами є силіцій (IV) оксиди різних модифікацій, які за рахунок розвиненої поверхні та активних силанольних груп на ній здатні до фізичної та хімічної взаємодії із полімерною матрицею. Для досліджень вибрані найбільш розповсюджені промислові марки силіцій (IV) оксидів: біла сажа, аеросил, силікагель. В результаті мікроскопічних досліджень встановили, що частинки силіцій (IV) оксидів марок «біла сажа» і «аеросил», як у вихідному вигляді так і у полімерній матриці активно агрегатуються, створюючи агломерати розмірами до десятків мкм. Таке явище не спостерігається при використанні силікагелю. Тому силіцій (IV) оксид марки «силікагель» доцільніше використовувати в якості наповнювачів для створення ПКМ конструкційного призначення, ніж інші матеріали, що досліджували. У роботі було приведено метод синтезу силікагелю із високим ступенем хімічної чистоти. Проведені порівняльні дослідження показників фізико-механічних, теплофізичних та триботехнічних властивостей конструкційного полімеру на основі ароматичного поліаміду фенілон С1 при наповненні промисловим та синтезованим силікагелями. Встановлено, що ПКМ із синтезованим силікагелем мають кращі властивості у порівнянні із промисловим. Це є наслідком того, що домішки, які присутні у промисловому силікагелі, зменшують його підсилюючий ефект на полімерну матрицю та негативно впливають на процес тертя при фрикційній взаємодії отриманого ПКМ із сталлю.

Ключові слова: силіцій (IV) оксид; силікагель; полімер; ароматичний поліамід; полімерний композиційний матеріал; фізико-механічні властивості

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*Corresponding author: e-mail address: Amber_UDHTU2@i.ua

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НАПОЛНИТЕЛИ НА ОСНОВЕ ДИОКСИДА КРЕМНИЯ ДЛЯ ПОЛИМЕРНЫХ КОМПОЗИЦИОННЫХ МАТЕРИАЛОВ КОНСТРУКЦИОННОГО НАЗНАЧЕНИЯ

Олег С. Кабат[1,2], Кристина В. Гети[3], Игорь Л. Коваленко[1], Анатолий Н. Дудка[1]
[1]Украинский государственный химико-технологический университет, пр. Гагарина, 8, Днепр, 49005, Украина
[2]Днепровский государственный аграрно-экономический университет, ул. Ефремова, 25, Днепр, 49600, Украина
[3]Днепровский национальный университет им. Олеся Гончара, пр. Гагарина, 72 Днепр, 49005, Украина

Аннотация

Проведен анализ по нахождению наполнителя для создания полимерных композиционных материалов (ПКМ) конструкционного назначения, который способен не только улучшить уровень свойств полимерной матрицы, а и значительно удешевить ее. Определено, что такими наполнителями являются диоксиды кремния разных модификаций, которые за счет развитой поверхности и активных силанольных групп на ней способны к физическому и химическому взаимодействию с полимерной матрицей. Для исследований были выбраны наиболее распространённые промышленные марки диоксида кремния: белая сажа, аэросил, силикагель. В результате микроскопических исследований установили, что частички диоксидов кремния марок «белая сажа» и «аэросил» как в исходном виде, так и в полимерной матрице активно агрегатируются, создавая агломераты до десятков микрометров. Такое явление не наблюдается при использовании силикагеля. Поэтому диоксид кремния марки «силикагель» целесообразнее использовать в качестве наполнителя для создания ПКМ конструкционного назначения, чем другие рассматриваемые материалы. В работе приведен метод синтеза силикагеля с высокой степенью химической чистоты. Проведены сравнительные исследования показателей физико-механических, теплофизических и трибологических свойств ПКМ конструкционного назначения на основе ароматического полиамида фенилона С1, наполненного промышленным и синтезированным силикагелем. Установлено, что ПКМ с синтезированным силикагелем имеют лучшие свойства в сравнении с промышленным. Это является следствием того, что примеси, которые находятся в промышленном силикагеле, уменьшают его усиливающее влияние на полимерную матрицу и негативно влияют на процесс трения при фрикционном взаимодействии полученного ПКМ со сталью.

Ключевые слова: диоксид кремния; силикагель; полимер; ароматический полиамид; полимерный композиционный материал; физико-механические свойства

Introduction

The modern development of science and technology is practically impossible without the use of new constructural materials with a unique combination of properties used to manufacture parts of machines and mechanisms operating at high levels of pressures and temperatures under the action of aggressive and abrasive media, alternating loads and others. Synthetic polymers are some of this kind of materials. Due to the unique combination of properties (low density, high level of chemical resistance and manufacturability in processing into products, high durability, ability to work in friction units without lubrication or with extreme lubrication, etc.), these materials are becoming more widely used in modern technology [1–5]. As disadvantages of polymers it should be noted, that they do not have the physical-mechanical and thermophysical properties of a sufficiently high level. For their improvement and directional regulation polymers are filled with materials of different nature and morphology [6–10], which act not only as components that strengthen the polymer matrix, but can also that significantly reduces the cost of PC products.

Silica of various modifications is one of those materials. [11; 12]. The use of such fillers is caused by their low cost (up to $5 per 1 kg), distribution in nature (more than 15 % of the earth's crust consists of this material) and the ability to physical (due to the developed surface) and chemical (due to active «silanol» groups on the surface) interaction with the molecules of the polymer during its processing into products [13; 14]. There are known PCs in which the introduction of silica of various modifications leads to a significant improvement in their physico-mechanical and thermophysical properties [15–18]. Therefore, the use of this filler to create new PCs with a unique combination of properties is an actual task.

Objects of research

The most common industrial brands of silica as aerosil, white soot, silica gel were selected for research. They differ in both morphology and chemical composition. The characteristics of the studied silica are shown in table 1.

Table 1

Characteristics of the studied silica [19–21]

Name	Brand	The average particle size, nm	Specific surface, m^2/g	Composition		
				SiO_2 (after piercing no less)	impurities (no more)	moisture (no more)
Aerosil	А-380	5–15	380 ± 20	99.9	0.1	1.5
White soot	БС-120	19–27	120 ± 20	90.5	3.0	6.5
Silica gel	КСКГ*	7–10 µm	-	91.5	3.0	5.5

* silica gel was ground to a size of 7–10 micron

Aromatic polyamide (copolymer of polymeta- and polyparaphenyleneisophthalamide), of the brand «phenylone C1», was selected as the matrix polymer of constructural purposes. Its structural formula is shown in Fig. 1.

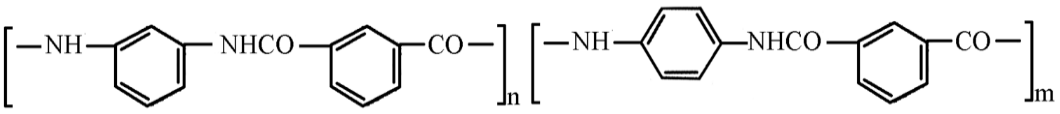


Fig. 1. The structural formula of phenylone C1

This polymer in its original form is a powder with a particle size of 20-40 mkm, which is processed into the product by the method of compression molding in preheated forms.

The goods from aromatic polyamides have a high level of physico-mechanical and thermophysical properties, but its cost is to $100 per 1 kg so they didn't get a wide spread in the industry.

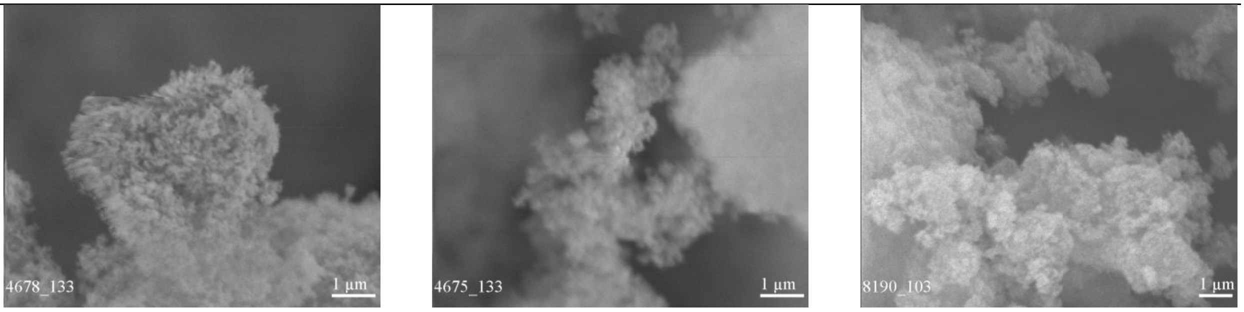
Research methodsThe study of the morphology of the silica particles and the surfaces of the cleavage of the PC was performed on electron (Superprobe-733 (Jeol)) and optical (MBR-1E) microscopes. The density of the materials was determined by hydrostatic weighing according to ISO 1183-1. The compressive stress at yield (σ_y) and the modulus of elasticity (E) were determined on a universal burst machine «2167 P-50» in accordance with ISO 604. The hardness (H) of the materials was determined by the method of indentation of the ball on the hardness tester «2013 ТШСП» in accordance with ISO-2039-1.

Vicat softening temperature T_{VC} was determined on the «FWV-633/10» according to ISO 1183-1. The coefficient of friction (f_{TP}), the temperature at the friction surface (T) and the intensity of linear wear (I_h) in the friction interaction of the developed PC with steel were determined by the machine «2070 CMT-1» in the friction mode without lubrication according to the disc-pad scheme. The steel sample of steel 45 with a roughness of Ra = 0.32 µm and a hardness of 45–50 HRC were used.

Experimental part

In modern industry the synthetic silica brands such as aerosil, white soot and silica gel have become widespread. They differ from each other by the method of production, morphology and chemical composition [15–17]. Aerosil is a pyrogenic colloidal silica (composed of at least 99.8 % SiO_2), which is obtained by flame hydrolysis of volatile silicon-containing substances. White soot is hydrated silica (consisting of not less than 85–95 % SiO_2, other impurities of oxides and hydroxides of iron, aluminum, magnesium, etc.), which is obtained from a precipitate formed by the action of acids on a solution of sodium silicate, followed by filtration, washing and drying. Silica gel is a dried silicic acid gel (comprising at least 85–95 % SiO_2, other impurities), which is obtained by reacting a solution of sodium silicate with the acid, followed by drying and washing of the product.

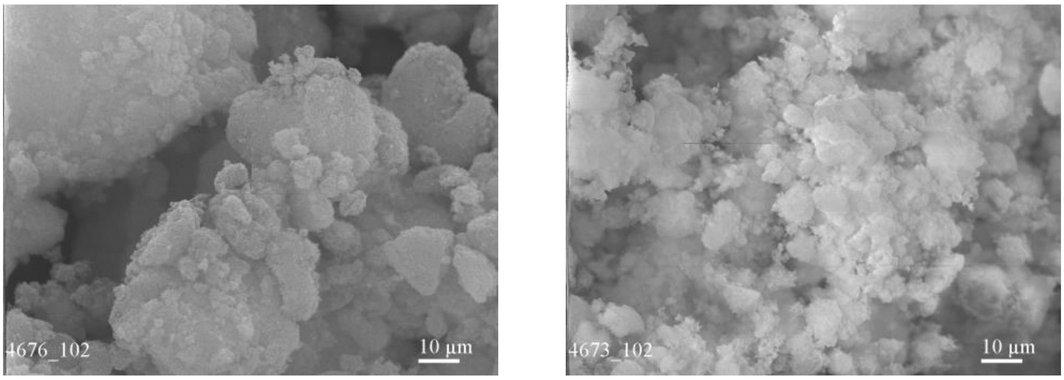
Different methods of producing synthetic silica determine their properties: size, shape of particles, presence or absence of pores, surface properties. All these materials have a sufficiently developed surface of up to 380 g/m^2 with a large number of pores, micro- and sub-micropores (Fig. 2), which contribute to the physical adsorption of polymer molecules during PC processing into products.



a b c

**Fig. 2. Micrographs of particle surfaces of silica grades:
a – aerosil; b – white soot; c – silica gel**

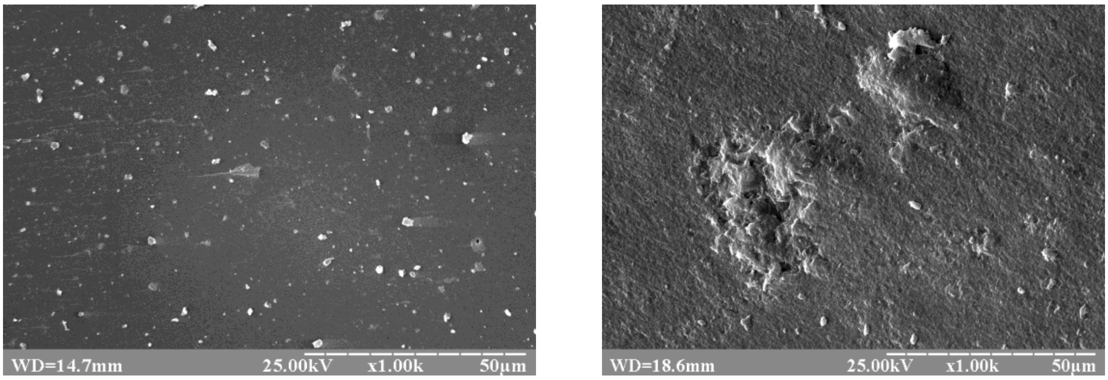
The size of the elementary particles of silica of such brands as aerosil and white soot are from 5 to 70 nm, which contributes to their active agglomeration. The size of the obtained agglomerates can be up to tens of microns (Fig. 3).



a b

**Fig. 3. Micrographs of agglomerates of silica brands:
a – aerosil; b – white soot**

When using these materials as fillers of polymers it is not possible to grind them to elementary particles. In the process of obtaining of the polymer composition and its processing into products it is possible to reduce the size of agglomerates of this silicas to 10–20 microns (Fig. 4).



a b

**Fig. 4. Micrographs of the surface of the cleavage of the original polymer (a)
and PC with white soot (b)**

However, considering that the silica agglomerates of aerosil and white soot are structures that are easily destroyed by loading, no significant enhancing effect of their use as a filler of polymer matrices has been obtained [22].

Silica gel in its original form is not often used as a filler for polymers due to the fact that its industrial species have a particle size of 0.1 to 10 mm. Therefore, for further studies, silica gel was ground on a disintegrator (Fig. 5a).

The particles of crushed silica gel in the original form are agglomerated, but in the joint processing with the polymer into the product almost all of these agglomerates are broken (Fig. 5b). Therefore, the use of silica gel as a filler of polymer matrices is the most appropriate of all considered synthetic silica. This fact is confirmed by the previous studies [23].

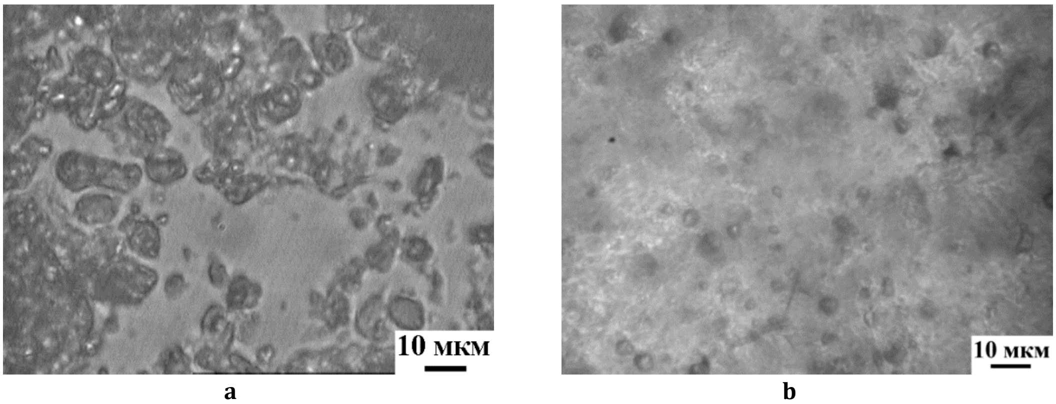


Fig. 5. Micrographs of crushed silica gel particles (a) and the surface of the PC chipped with this filler (b)

However, silica gel produced by industry is not silica with a high level of chemical purity, because it has up to 10 % impurities (oxides, hydroxides, salts, etc.), which can affect the level of PC properties. Therefore, it is interesting to determine the effect of high purity «silica gel» on the polymer matrix and to study the level of physico-mechanical, thermophysical and tribotechnical properties of PC based on them.

The choice of the products and the mode of synthesis of silica gel was carried out in accordance with the previous studies [24] in order to obtain a filler with the maximum developed specific surface area and the maximum number of active «silanol» groups on it.

The main ingredient for producing silica gel with high chemical purity is soluble sodium silicate, which according to «ГОСТ Р 50418-92» has up to 1 % impurities. Distilled water and the aqueous solution of hydrochloric acid (ч.д.а., ГОСТ 3118-77) were also used.

The technology of obtaining silica gel with a high degree of chemical purity was as follows:

- obtaining the hydrogen solution of sodium silicate;
- acidification with aqueous solution of hydrochloric acid to transition of the resulting system to a gel state, by the reaction:

$Na_2SiO_3 + 2HCL \rightarrow SiO_2 + 2NaCl + H_2O$;

- drying, grinding and washing of the resulting gel.

The concentrated hydrogen solution of sodium silicate is obtained by combining it with distilled water at a temperature of 50–55 °C. The resulting solution has up to 20 % Na_2SiO_3 (based on solid product) in water.

Its acidification occurs with 10 % aqueous hydrochloric acid solution, which is fed to the reactor by droplets with constant stirring of the reaction medium. Upon reaching the pH of the resulting solution 5.5 the reaction medium goes into a gel state.

Drying of the gel occurs in a vacuum oven at a temperature of 40–60 °C. It lasts until the complete transition of the gel to a solid state promotes, and stops when the weight loss of the obtained product ends under these conditions. The obtained solid product is ground on a disintegrator according to the method used to obtain a filler from industrial silica gel. The shredded system is washed in distilled heated water to 70 °C for 10 minutes. This allows the reaction by-products (NaCl salt and acid residues) to be washed out of the solids.

The obtained silica of the grade silica gel has a high degree of chemical purity, its particles have a small size and a well-developed surface that allows to use this material as a filler for polymer matrices.

To determine the basic size of the synthesized silica gel it was used a differential histogram of particle size distribution, which was created using micrographs obtained on an electron scanning microscope (Fig. 6). Based on the fact that the particles of silica gel have a spherical shape, their main calculated size was taken by the diameter. From the obtained histograms is was determined that the largest number of particles (60 %) accounts for the size range from 5 to 10 microns. That is, it can be argued that the main particle size of the obtained "silica gel" lies in this range, which allows to use this material as a filler for polymer matrices.

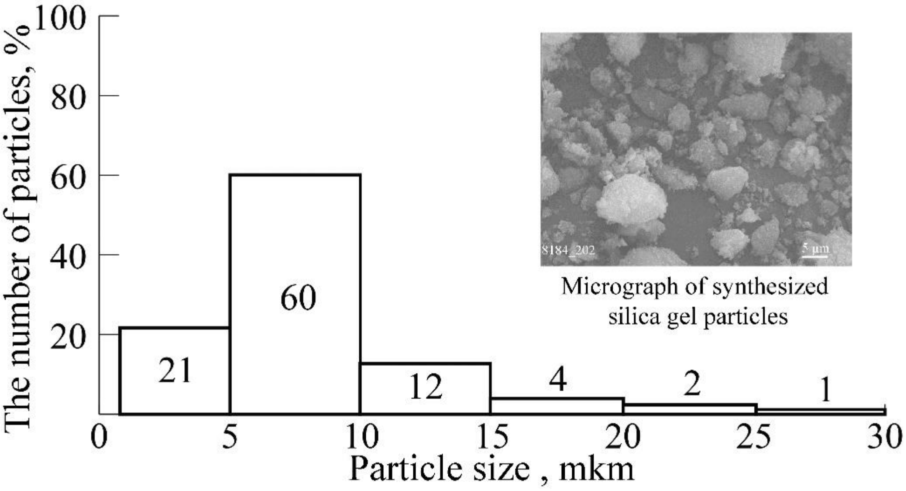


Fig. 6. Histogram of particle size distribution of the synthesized silica gel

From the obtained histograms is was determined that the largest number of particles (60 %) accounts for the size range from 5 to 10 microns. That is, it can be argued that the main particle size of the obtained "silica gel" lies in this range, which allows to use this material as a filler for polymer matrices.

For further studies of the effect of silica gel on the properties of polymer matrices, aromatic polyamide was selected, which belongs to plastics of constructural designation and approaches the level of its properties to low carbon steels [25]. This polymer was filled with industrial and synthesized silica gel. Comparative analysis of indicators of physico-mechanical, thermophysical and tribotechnical properties of the obtained PC is shown in table 2.

Table 2

Physico-mechanical, thermophysical and tribotechnical properties of PC based on aromatic polyamide and silica gel

Properties	The content of silica gel in the PC							
	industrial				synthesized			
	5	10	20	30	5	10	20	30
Density, kg/m^3	1350	1380	1405	1411	1336	1340	1350	1365
Compressive stress at yield σ_y, MPa	242	245	257	245	242	243	266	264
The modulus of elasticity E, MPa	3105	3216	3289	3280	3210	3333	3370	3362
Hardness H, Mpa	219	234	192	183	220	240	200	180
Vicat softening temperature T_{VC}, ^oC	272	273	281	285	271	273	283	288
Coefficient of friction f_{TP}	0.33	0.29	0.31	0.33	0.31	0.29	0.30	0.31
Temperature at the friction surface T, ^oC	98	92	97	106	90	81	82	84
Intensity of linear wear $I_h \times 10^{-9}$, m/m	57	25	37	62	40	19	34	49

From the results obtained we can conclude that the level of physico-mechanical and thermophysical properties of PC with industrial and synthesized silica gel has no significant differences. Higher density in PC with industrial silica gel is caused by the presence of impurities (oxides, metal hydroxides, etc.) in this filler. The compressive stress at yield and modulus of elasticity, hardness, and Vicat softening temperature in PC with synthesized filler are 5-7% better than in the materials with industrial silica gel. This is due to the better enhancing effect of the synthesized silica gel on the polymer matrix, which is associated with both the physical and chemical interaction of these components of the PC. It should be noted that the tribotechnical properties of PC with synthesized silica gel are much better than with the industrial ones. This is due to the fact that this filler has less impurities, which in this case adversely affect the friction and wear of the PC during the friction interaction with steel.

It should be noted, that silica gel is 20 times cheaper then aromatic polyamide. It contributes to significant reduction of cost of the obtained PC compared to the initial polymer.

Conclusions

As a result of the work, it was determined that silica is one of the most progressive fillers for PC for constructional purposes, which can not only improve the level of properties of the polymer matrices, but also reduces the cost of manufacturing parts from them.

It was obtained that silica gel has the best reinforcing effect on the polymer matrix among the most common silica, produced by the industry.s It does not create agglomerates that promote to destraction of PC at high loadings, like silica of the brands aerosil and white soot. The technique of synthesis of silica gel with high level of chemical purity is presented.

A comparative analysis of the physico-mechanical, thermophysical and tribotechnical indices of the PC for constructural purposes, based on aromatic polyamide filled with industrial and synthesized silica gel was carried out.

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