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A new type of bentonite-based non-woven composite

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Abstract: Sandwich-like composites based on clays and textiles are extensively applied in various fields, including civil engineering and environmental protection. In this paper, the synthesis of a new type of composite with embedded bentonite particles within a non-woven polyester matrix is presented. The synthesized composite has improved mechanical properties compared to the corresponding non-woven matrix. Although more than two-times thinner, the synthesized composite showed mechanical properties similar to those of a commercial composite chosen for comparison. Sorption test results confirmed that the contribution of the textile component to the sorption of aqueous Cu(II) ions by the composite was negligible. The sorption of aqueous Cu(II) ions on the synthesized composite was best-fitted using the Langmuir model. The presented study confirmed that the loss of bentonite particles from the composite can be eliminated using the suggested synthesis method.

Keywords: composite; bentonite; non-woven; mechanical properties; sorption.

INTRODUCTION

Bentonites are clays rich in smectite but also containing various associated minerals, such as carbonates, feldspars, quartz, *etc.*^{1–3} Due to their high smectite content, bentonites are good sorbents. Numerous investigations were focused on bentonite as a sorbent of inorganic pollutants, particularly of toxic aqueous metal ions.^{4–7} When bentonite is utilized as sorbent, a problem exists of its separation after use. The fineness of the particles and their tendency to swell impede the separation process. This increases the cost of the employment of bentonite as a

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sorbent. One of the possible ways to overcome this is the use of bentonite powder firmly fixed to an inert support.

On the other hand, composites based on clays and textiles are materials that lately have been extensively applied in civil engineering and environmental protection.^{8,9} Commercial composites are most commonly applied as geosynthetic clay barriers. They are produced in a variety of forms. They consist of a clay layer (typically bentonite) bonded to a layer or layers of a geosynthetic material. The geosynthetic materials are either geotextiles or geomembranes. Geotextile-based composites have a sandwich-like structure. They are bonded with an adhesive, needle-punching, stitch-bonding or a combination of these methods. In geomembrane-supported composites, the bentonite is bonded to the geomembrane using an adhesive.^{8,9} The main forms of commercial composites are schematically presented in Fig. 1.

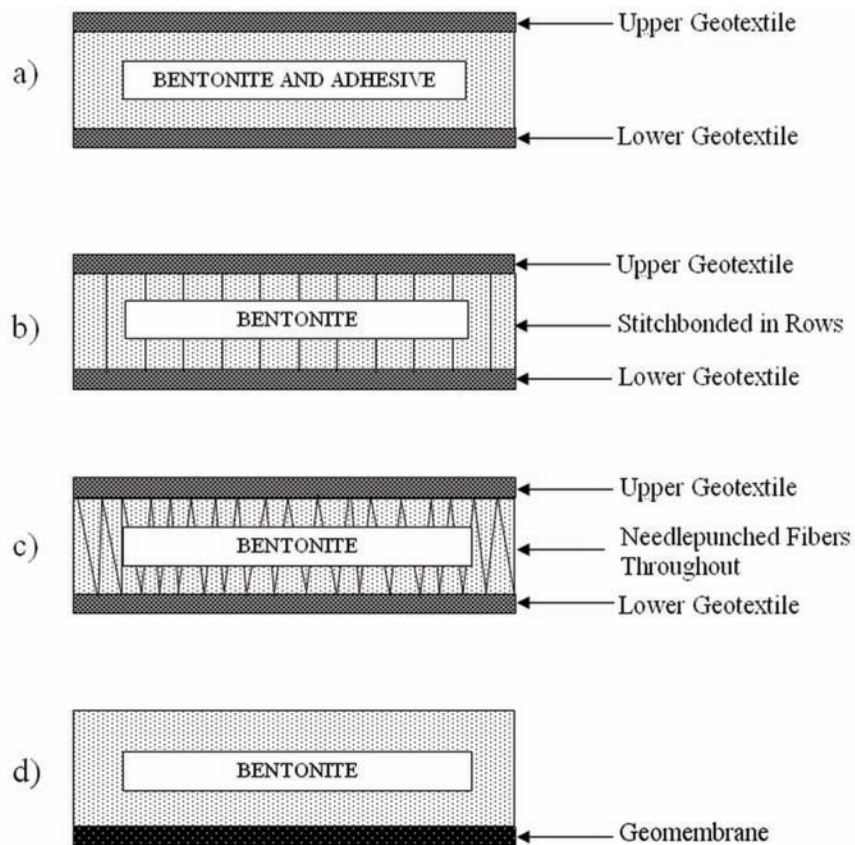


Fig. 1. Forms of bentonite-textile composites: a) adhesive-bonded sandwich structure; b) stitch-bonded sandwich structure; c) needle-punched sandwich structure; d) bentonite adhered to geomembrane (two-layered structure).

Geotextile composites primary differ in the form of the bentonite, type of fiber, type of textile and the bonding methods used to bind the composite. In composite synthesis either the sodium or calcium type of bentonite can be used in powdered or granular form. The type of polymer fiber and their fineness may differ in the composites. Both woven and non-woven textiles are used as geotextiles. Often, the lower geotextile layer is woven, while the upper one is non-woven.^{8,9} Although the overall configuration of the composite affects its performance, the primary factors are clay quality, the amount of clay used per unit area and its uniformity.

One of the drawbacks of composites with a sandwich-like structure is the insufficiently firm fixation of the bentonite layer to the textile. For example, when rolls of these materials are employed, powder is easily displaced, both laterally within the product and through the geotextile. Although bentonite is non-toxic and can even be used as a pharmaceutical,¹⁰ bentonite powder is considered to be a respiratory hazard due to the inhalable crystalline quartz and fine smectite particles.¹¹ Finally, the migration of the powder within the composites with a sandwich-like structure causes localized inconsistencies in the mass per unit area of the product, which in turn creates variability in its hydraulic performance.^{12,13}

In this paper, a new type of composite is presented, synthesized with bentonite particles embedded within a non-woven polyester matrix by applying bentonite in the form of a suspension. The goal was to obtain a composite with a more consolidated structure and avoid losses of the bentonite powder from the composite.

The sorptive properties of textile-bentonite composites have received less attention in the literature¹⁴⁻¹⁶ in comparison to bentonite alone.^{1,4-7,17-20} In the presented investigations more attention was focused on the sorptive properties of the synthesized composite. Removal of aqueous Cu(II) ions was chosen as a model system. Toxic metals, such as lead, copper, cadmium, zinc and nickel, are among the most common pollutants found in industrial effluents.²¹ Of the 71 priority pollutants analyzed, copper, was most frequently detected, along with lead and zinc.²² The increased content of copper in the liver, brain, and other tissues leads to hepatitis, kidney dysfunction, brain disorders, and other problems.^{22,23} According to the Environmental Protection Agency (EPA), the maximum allowed amount for copper in discharged water is 1.3 mg dm^{-3} .²²

The objective of the investigations presented herein was to synthesize a composite with bentonite particles fixed within a non-woven textile structure to prevent bentonite losses. Further investigations presented in this paper were performed in order to prove that the synthesis conditions do not impair the sorptive properties of bentonite and the mechanical properties of non-wovens.

EXPERIMENTAL

The composite was synthesized using bentonite and commercial materials. Needle-punched polyester non-woven fabric (M-PROINTEX, Mladenovac, Serbia) with a nominal mass per unit area $m_A = 300 \text{ g m}^{-2}$ and a thickness of $\delta = 3 \text{ mm}$ was used as the non-woven matrix. A carboxymethyl cellulose-based adhesive (Aero Balkan, Belgrade, Serbia) solution was applied to fix the bentonite particles to the non-woven (NW) material. The mineral constituent was bentonite powder ($< 74 \mu\text{m}$) from the coal and bentonite mine "Bogovina", Serbia. The chemical, physical, morphological and textural properties of bentonite powder were presented previously.^{24,25} The chemical composition of bentonite dried at $110 \text{ }^\circ\text{C}$ (given in mass %) was: SiO_2 , 57.51; Al_2O_3 , 17.13; Fe_2O_3 , 7.67; MgO , 2.35; CaO , 1.81; Na_2O , 0.75; K_2O , 1.18. XRD analysis confirmed smectite as the dominant phase with quartz as the major associated mineral. Small amounts of illite, calcite, feldspar and amorphous material were also detected. The specific surface area of the bentonite powder was calculated according to the Brunauer, Emmett, Teller method from nitrogen adsorption-desorption isotherms and the obtained value was $96 \text{ m}^2 \text{ g}^{-1}$.

The following synthesis procedure was used. First the adhesive was dissolved in distilled water at $35 \text{ }^\circ\text{C}$ and stirred until a homogeneous solution was obtained. Bentonite powder was then dispersed in the aqueous solution of the adhesive. The dispersion was stirred to form a homogeneous suspension.

The viscosity of the bentonite suspension in an adhesive solution was significantly greater than the viscosity of either the adhesive solution or a dispersion of bentonite in pure water. The suspension viscosity of the dispersion affects the ability of the suspension to penetrate the NW material. If the suspension viscosity is higher than optimal, the suspension is unable to pass through the entire NW fabric and a layer of bentonite is formed on the top of it. On the other hand, if the suspension viscosity is lower than optimal, the suspension passes through the NW fabric and the bentonite does not remain within the NW material. A suspension containing 50 g dm^{-3} bentonite dispersed in a solution of adhesive having a concentration of 6.7 g dm^{-3} was found to have the optimal viscosity. The viscosity of this suspension was measured using a Brookfield Eng. Lab. Inc, USA viscometer.

The synthesis of the composite was performed by applying the suspension with the optimal viscosity onto the NW surface. Water from the wet composite was removed by drying to constant mass in an oven at $60 \text{ }^\circ\text{C}$. In the next step, the bentonite suspension was applied once more, but this time on the opposite side of the textile. Drying of composite at $60 \text{ }^\circ\text{C}$ was performed again until a constant mass was reached.

In order to characterize the obtained composite, the standard methods for geosynthetic investigation were used. The British Standard EN 14196 was used for the determination of the mass per unit area²⁶ and the International standard ISO 9863-1 for the measurement of the composite thickness.²⁷

Load per unit length and elongation were determined using a Wykeham Farrance mechanical testing machine (BR-IPGM-KID-03) according to the ISO 10319 test method.²⁸ The experiments were performed on the original NW fabric and the corresponding composite. The materials were examined both in the machine direction (md) and the cross machine direction (cmd).

Scanning electron microscopy (SEM) photos of the synthesized and commercial composite were obtained using an SEM JEOL JSM-6610LV.

There is no standard method to estimate the loss of bentonite powder from a composite. In this study, two different tests, a more and a less severe one, were adopted. Their purpose

was to show firmer incorporation of the bentonite particles within the synthesized composite structure in comparison to a commercial composite with a sandwich-like structure Bentofix BFG 5000.²⁹

The first type of test was performed with a rotating tube attrition tester – the Spence method.³⁰ Samples were loaded into cylindrical tubes placed on a disc-shaped frame. The frame was rotated at a speed of 25 rpm for two hours. For the second test, the samples were placed in an ultrasonic bath (Selecta Ultrasound-H), operating at a frequency of 40 kHz for 30 min. The mass of the samples was measured before and after each test by both methods. The results are presented as mass loss in percents (χ). For each method, 10 samples of the commercial and of the synthesized composite were used. The average values and standard deviation were calculated.

In order to determine the contribution of each constituent of composite to the sorption of aqueous Cu(II) ions, batch experiments were performed separately on: a) NW fabric impregnated with adhesive – the control textile sample; b) bentonite powder and c) the studied composite. The masses of the samples were: 0.25 g for bentonite powder and the control textile, while the composite mass was 0.5 g, containing approx. 0.25 g of NW fabric and 0.25 g of bentonite. The sorption test was performed with 50 cm³ of aqueous solution of Cu(II) nitrate with the initial Cu(II) concentration, c_0 , of 5×10^{-3} mol dm⁻³.

When the contribution of each constituent of the composite to the sorptive properties of the composite had been determined, an isothermal study on the synthesized composite only was performed. All experimental conditions were the same as in the previous test except the initial concentration of aqueous Cu(II) ions was varied within the range 2.5×10^{-3} – 10.0×10^{-3} mol dm⁻³.

In all sorption experiments, the samples were shaken at 20 ± 2 °C in a temperature-controlled water bath shaker (Mettmert WNE 14 and SV 1422). Measurements were performed after predetermined periods (0.25, 0.5, 1, 2, 4, 12 and 24 h). When bentonite was used as the sorbent, the bentonite suspensions were centrifuged at 5000 rpm for 15 min (Hettich EBA-21 to separate the sorbent from the dispersion).

The concentrations of Cu(II) ions before and after the sorption test were analyzed using a Thermo Electron Nicolet Evolution 500 UV–Vis spectrophotometer. The absorption peak at 806 nm, corresponding to Cu(II), was chosen for the estimation of the Cu(II) concentration, since a linear dependence of absorbance on Cu(II) concentration was found to have a correlation coefficient (R) of 0.999.

The Cu(II) uptake (p) given in percents was calculated as follows:

$$p = 100 \frac{c_0 - c_t}{c_0} \quad (1)$$

where c_0 is the initial Cu(II) concentration (mg dm⁻³) and c_t is the Cu(II) concentration in solution after time t .

The amount of the aqueous Cu(II) ions sorbed by the sorbent (composite) at the equilibrium q_e (mg g⁻¹) was calculated by the following mass-balance relationship:

$$q_e = \frac{V(c_0 - c_e)}{W} \quad (2)$$

where c_e is the equilibrium Cu(II) concentration, V is the volume of the solution (dm³) and W is the mass of the sorbent (g).

RESULTS AND DISCUSSION

A mass per unit area of the synthesized composite of $m_A = 590 \pm 20 \text{ g m}^{-2}$ and a thickness of $\delta = 2.89 \pm 0.18 \text{ mm}$ were established according to standard methods.^{26,27} The bentonite content in the composite was determined to be 51 ± 2 mass %.

The viscosity of the suspension with the optimal composition was measured using a Brookfield viscometer. The dependence of the viscosity on the shear rate is given in Fig. 2.

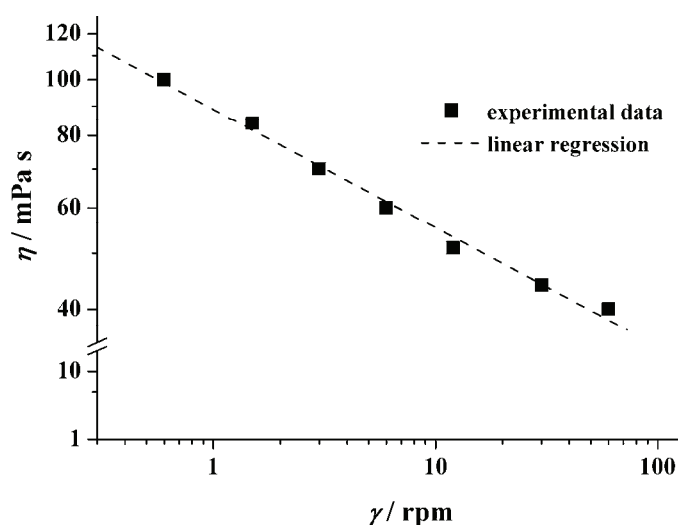


Fig. 2. Dependence of the viscosity of a bentonite/adhesive suspension on shear rate.

The obtained linear regression can be represented by the following equation:

$$\log \eta = 1.948 - 0.205 \gamma \quad (3)$$

where: η – viscosity, $\gamma = dv/dx$ – shear rate, with $R = 0.9959$.

As expected, the viscosity of the bentonite/adhesive suspension obeyed a “power law” and exhibited a behavior characteristic for pseudoplastic fluids.³¹

Since the synthesis of the new type of composite presented in this paper can be performed using bentonites and adhesives of different origin, it is useful to have a synthesis condition figure of merit independent of the origin of the applied raw materials. The use of suspension with a viscosity given in Eq. (3) enables the given synthesis process to be extended to a variety of raw materials.

The SEM microphotographs of the synthesized composite and the commercial Bentofix BFG 5000 with a sandwich-like structure²⁹ are presented in Fig. 3. Bentonite particles firmly embedded within the NW structure of the synthesized composite are clearly visible in Figs. 3a–3b. The particles completely surround the textile fibers and remain connected even in the cases of severe bending and

cutting of the composite during the sample preparation for electron microscopy. On the other hand, the integrity of structure was destroyed during an identical preparation of a commercial sample for SEM. In Fig. 3c only textile fibers relatively loosely in contact with the surrounding bentonite powder are visible.

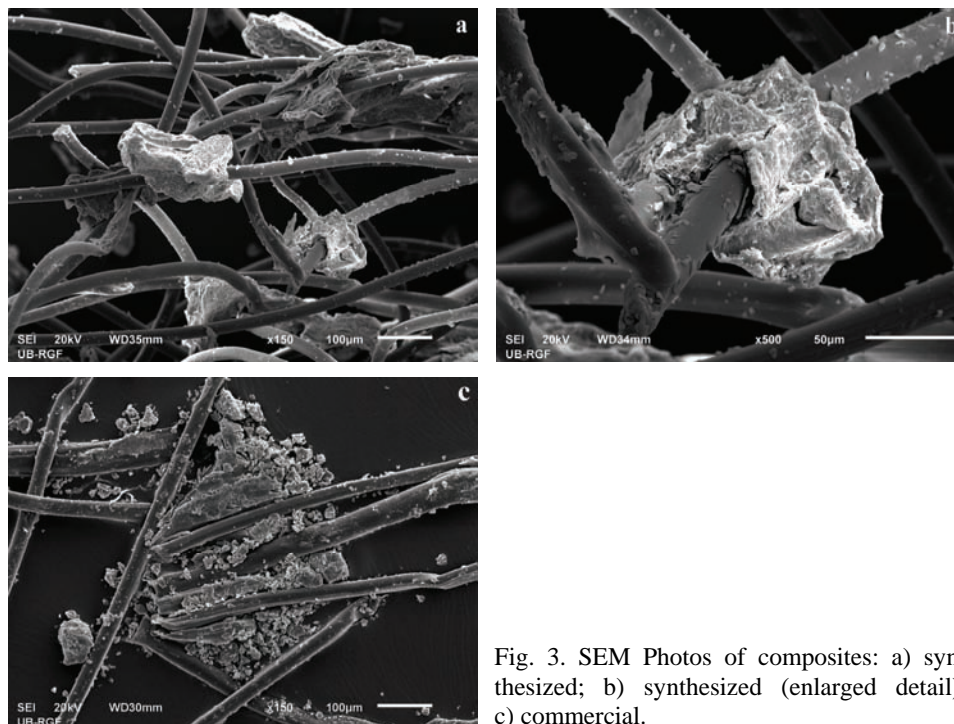


Fig. 3. SEM Photos of composites: a) synthesized; b) synthesized (enlarged detail); c) commercial.

The recorded load per unit length (σ) vs. relative elongation (ε) curves are presented in Fig. 4 for both the NW fabric and the synthesized composite. The obtained mechanical properties are given in Table I.

From the results presented in Fig. 4 and Table I, it can be concluded that the load per unit length at break (σ_{\max}) was higher, while the relative elongation at break (ε_b) was lower in the machine direction than in the cross machine direction for both the NW fabric and the corresponding composite. The composite formation improved the load per unit length and lowered the elongation of the starting non-woven fabric in both directions by approx. 20 %. In addition, it was also necessary to apply 8, 16 and 28 times greater force to the composite than to the corresponding NW in order to achieve elongations of 10, 5 and 2 %, respectively. These data show to what extent the mechanical properties of the composite are improved compared to the corresponding non-woven fabric, especially at lower loadings.

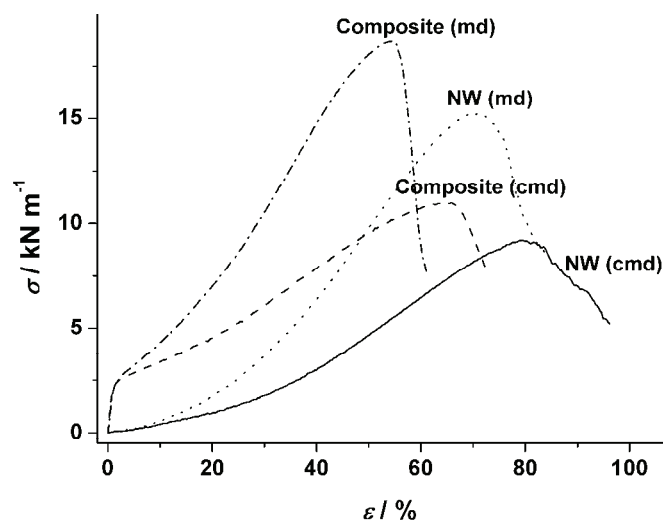


Fig. 4. Load per unit length vs. relative elongation for the NW fabric and the synthesized composite.

TABLE I. Load per unit length and elongation of the composite and the non-woven fabric (σ_{\max} – load per unit length at break, ϵ_x – relative elongation at break, md – machine direction, cmd – cross machine direction)

Sample	$\sigma_{\max} / \text{kN m}^{-1}$	$\epsilon_x / \%$	$\sigma / \text{kN m}^{-1}$ at $\epsilon = 2 \%$	$\sigma / \text{kN m}^{-1}$ at $\epsilon = 5 \%$	$\sigma / \text{kN m}^{-1}$ at $\epsilon = 10 \%$
NW(md)	15	71	0.089	0.222	0.566
Composite (md)	19	55	2.510	3.201	4.309
NW(cmd)	9	79	0.086	0.163	0.412
Composite (cmd)	11	66	2.440	2.830	3.367

Photographs of fractures of the composites after the mechanical tests are presented in Fig. 5 for comparison.

It can be observed that upon rupture, the bentonite phase remains firmly fixed within the synthesized composite, while there is a significant release of the clay from the commercial one.

The results for the mass loss obtained using both the rotating tube attrition tester and ultrasound test are given in Table II for the synthesized composite and for one of the most common commercial composites with a sandwich structure, Bentofix BFG 5000.²⁹ In addition to the data for bentonite loss, Table II contains some physical and mechanical properties of the synthesized and commercial composites.

The loads per unit length required for the rupture of both composites were very similar. The test samples were of equal length and width but the commercial composite was more than two-times thicker than the synthesized one. This implies better mechanical properties of the synthesized material.

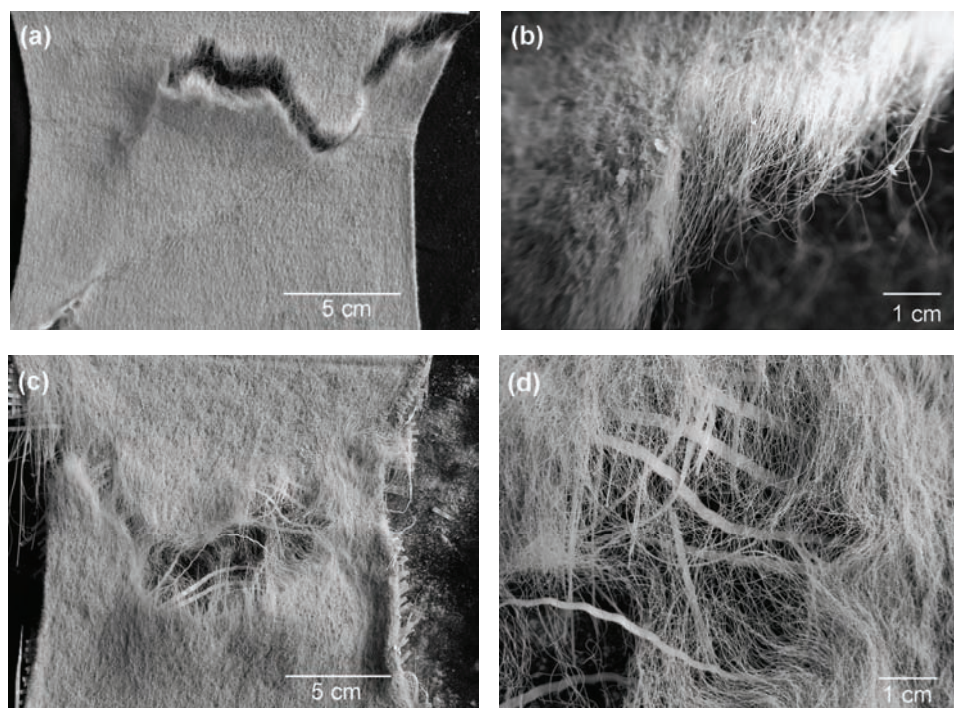


Fig. 5. Photographs of samples after mechanical tests: a) synthesized, b) synthesized (enlarged detail), c) commercial and d) commercial (enlarged detail).

TABLE II. Comparison of the properties of the synthesized and the commercial composite (δ – thickness, m_A – nominal mass per unit area, σ_{\max} – load per unit length at break, ε_x – relative elongation at break, md – machine direction, cmd – cross machine direction, χ – mass loss)

Sample	δ / mm	m_A / g m ⁻²	σ_{\max} / kN m ⁻¹		ε_x / %		Rotating test tube		Ultrasound bath test	
			md	cmd	md	cmd	χ / %	Std.	χ / %	Std.
Commercial	7	5500	20	11	10	5	70	3	4.34	1.1
Synthesized	3	600	19	11	55	66	5	2	0.73	0.1

Table II shows that the mass loss after the performed rotating test tube experiment was 14 times greater for the commercial than for the synthesized composite. In the ultrasonic test, the mass loss was less expressed; however, it was still 6 times greater for the commercial composite than for the synthesized one. Therefore, the most important advantage of the synthesized composite is its compact structure that simultaneously enables a firm fixation of bentonite within the composite matrix.

The sorption data for the relative uptake of aqueous Cu(II) ions on the bentonite, non-woven fabric impregnated with adhesive (control textile sample) and the composite vs. contact time are presented in Fig. 6.

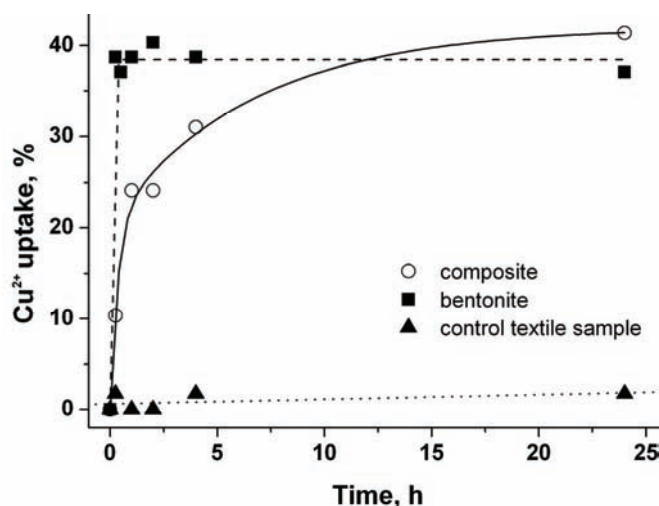


Fig. 6. Kinetics of Cu(II) uptake by the investigated samples; $c_0 = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$.

The data presented in Fig. 6 show that the sorption of aqueous Cu(II) ions by the NW fabric was negligible in comparison to that of bentonite and the composite and that the entire sorption capacity of the composite can be attributed to bentonite. The uptake of aqueous Cu(II) ions by bentonite was faster than by the composite, since in the latter, the bentonite particles were incorporated in the NW fabric and it was more difficult for the aqueous Cu(II) ions to reach the bentonite. For longer sorption times, the composite reached the sorption capacity of pure bentonite. The presence of adhesive did not impair the sorptive properties of bentonite in the composite, *i.e.*, the bentonite did not lose its sorptive properties during composite synthesis.

The sorption data for the uptake of aqueous Cu(II) ions by the composite *vs.* contact time at different initial concentrations are presented in Fig. 7. The pH of the solution after sorption was 6, which is a pH value where no precipitation of Cu(II) occurs and therefore the decrease of the Cu(II) concentration can be ascribed solely to the sorption process.

The percentile uptake of aqueous Cu(II) ions by the composite decreased with increasing initial Cu(II) concentration, but the amount of sorbed ions increased. More useful data can be obtained by analyzing the equilibrium conditions. The equilibrium contact time was estimated to be 12 h, since further sorption was negligible after this time. The equilibrium data, commonly known as sorption isotherms, are the basic requirement for the design of sorption systems. These data provide information on the capacity of the sorbent or the amount required for the removal of a unit mass of sorbate under the system conditions.

The sorption isotherms obtained for different initial Cu(II) concentrations were applied to fit the models of Freundlich³² and Langmuir.³³

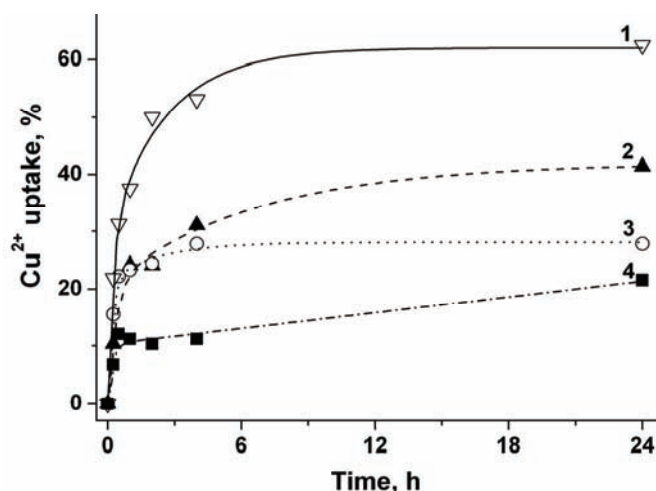


Fig. 7. Influence of contact time and initial Cu(II) concentration (c_0) on the uptake of Cu(II) by the composite (1 – $c_0 = 2.5 \times 10^{-3} \text{ mol dm}^{-3}$; 2 – $c_0 = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$; 3 – $c_0 = 7.5 \times 10^{-3} \text{ mol dm}^{-3}$; 4 – $c_0 = 10.0 \times 10^{-3} \text{ mol dm}^{-3}$).

The linear form of the Freundlich isotherm is represented by Eq. (4):

$$\ln q_e = \ln K_F + \frac{1}{n} \ln c_e \quad (4)$$

where K_F ($\text{dm}^3 \text{ g}^{-1}$) and n are the Freundlich adsorption constants characteristic for the system; they describe the adsorption capacity and adsorption intensity, respectively.

A plot of $\ln q_e$ vs. $\ln c_e$ gives a straight line with the slope and intercept equal to $1/n$ and $\ln K_F$, respectively (Fig. 8).

The Langmuir Equation can be expressed as follows:

$$\frac{c_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{c_e}{q_{\max}} \quad (5)$$

where q_{\max} is the maximum adsorption capacity of the sorbent (mg g^{-1}) and K_L is the adsorption constant ($\text{dm}^3 \text{ g}^{-1}$).

A plot of the specific adsorption (c_e/q_e) vs. the equilibrium concentration (c_e) gives a straight line with slope $1/q_{\max}$ and intercept $1/(q_{\max} K_L)$ (Fig. 9). The constants and correlation coefficients corresponding to both models are given in Table III.

According to the results presented in Table III, both models can be applied to the experimental data, but the correlation coefficient for the Langmuir model was closer to unity, hence this model was more appropriate. The validity of Langmuir model indicated the formation of a monolayer coverage of aqueous Cu(II) ions at the composite surface containing a finite number of homogenous

sorption sites. It also suggests that all adsorption sites were equivalent and there was no interaction between the adsorbed molecules.³⁴

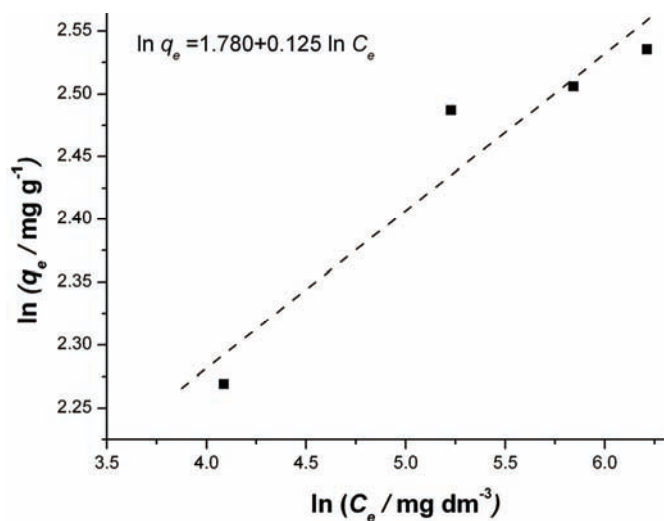


Fig. 8. Freundlich isotherm for Cu(II) sorption onto the composite.

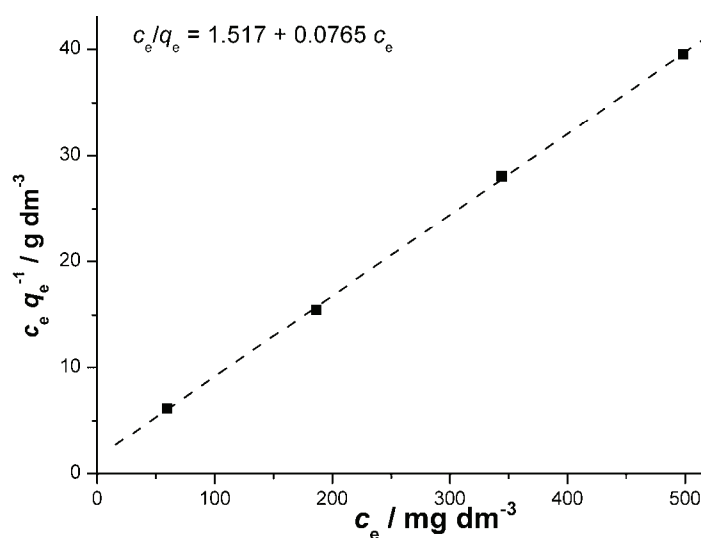


Fig. 9. Langmuir isotherm for Cu(II) sorption onto the composite.

TABLE III. Calculated sorption model constants and correlation coefficients

Freundlich constants			Langmuir constants		
$K_F / \text{dm}^3 \text{g}^{-1}$	n	R	$q_{\text{max}} / \text{mg g}^{-1}$	$K_L / \text{dm}^3 \text{g}^{-1}$	R
5.932	7.982	0.9560	13.09	50.36	0.9999

CONCLUSIONS

A new type of composite with bentonite particles embedded in non-woven textile was synthesized by applying bentonite in the form of suspension onto a non-woven fabric. The optimal viscosity of the bentonite suspension in the adhesive solution that enables appropriate composite formation was established. The composite formation improved the load per unit length and lowered the elongation of the starting non-woven fabric in both the machine and cross-machine direction. Although more than two-times thinner, the synthesized composite showed mechanical resistance similar to that of the commercial composite chosen for comparison.

It was proven that the problem of loss of bentonite particles from the composite structure was greatly reduced with the obtained composite. Both the rotating tube method and the ultrasonic test resulted in much lower mass loss values for the synthesized composite in comparison to the commercial composite.

The sorptive study showed that the contribution of non-woven component to the sorption of aqueous Cu(II) ions by the composite was negligible. The entire sorption capacity of the composite could be attributed to the bentonite. The bentonite did not lose its sorptive properties during the synthesis process. The isothermal data for Cu(II) sorption onto the composite were best fitted using the Langmuir model.

The obtained composite exhibited a compact structure with firm fixation of the bentonite within the composite matrix, as well as good mechanical and sorption properties. It may be regarded as a promising material for various applications in civil engineering and environmental protection, for example as geosynthetic clay barriers and in wastewater purification.

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ИЗВОД

НОВИ ТИП КОМПОЗИТА НА БАЗИ БЕНТОНИТА И
НЕТКАНОГ ТЕКСТИЛНОГ МАТЕРИЈАЛА

АЛЕКСАНДРА МИЛУТИНОВИЋ-НИКОЛИЋ¹, ЈАСМИНА ДОСТАНИЋ¹, ПРЕДРАГ БАНКОВИЋ¹, НАТАША
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Композити на бази глина и текстила са „сендвич“ структуром имају примену у различитим областима, укључујући грађевинарство и заштиту животне средине. У овом раду синтетисан је нови тип композита са честицама бентонита уграђеним у неткану полиестарску матрицу. Синтетисани композит има побољшана механичка својства у поређењу са одговарајућом нетканом матрицом. Иако два пута тањи од комерцијалног, са којим је упоређиван, синтетисани композит је показао слична механичка својства. Сорпционим

испитивањима потврђено је да текстилна компонента занемарљиво утиче на способност композита да сорбује Cu(II) јоне. Сорпција Cu(II) јона на синтетисаном композиту најбоље се описује Ленгмировим моделом. У овом раду је потврђено да се коришћењем предложене методе синтезе композита елиминише проблем испадања честица бентонита из композита.

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