

## The possibilities of using low-cost fibrous natural materials as sorbents for removing aliphatic hydrocarbons (C<sub>6</sub>–C<sub>15</sub>) from an aqueous solution

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### ABSTRACT

Sorption of oil-related products (including mainly the propellants) is the basic process that counteracts spreading these types of pollution into the environment. Plenty of synthetic substances (including multi-component petroleum products) must be removed both from the fresh and groundwater. The aim of this study was to compare the possibilities of using natural fibrous materials (also weed or waste materials): broadleaf cattail (*Typha latifolia* L.) seeds, peat and coconut fiber as sorbents of aliphatic hydrocarbons from an aqueous solution. In order to increase sorptive capacity, tested materials were mercerized in hot (80°C) NaOH for sorption properties improvement. The removal of aliphatic hydrocarbons (C<sub>6</sub>–C<sub>15</sub>) dissolved/emulsified in water was carried out by the "batch method". Conducted experiments have shown a low sorption level of the analyzed pollutants by *Typha* wool-seeds and medium level for coconut fiber. The best sorption behavior of hydrocarbons from water solution appeared for commercial peat sorbent which has been used as a control sample and the weakest for broadleaf seeds. On the other hand, the total sorption capacity of free hydrocarbons was obtained for coconut fiber and was almost 2 times lower than in the case of peat, but the best capacity was noted for broadleaf cattail seeds. Used natural fibrous materials appeared as sorbents with low (*Typha*) and average (coconut) effective sorbents, but (except for peat) environmentally friendly and economic for hydrocarbons dissolved or suspended in water sorption.

**Keywords:** Sorption; Hydrocarbons; Broadleaf cattail seeds; Peat; Coconut fiber; Mercerization

### 1. Introduction

In order to limit the negative effects of xenobiotic substances such as diesel, mazout or gasoline leaks, the handling of tank trucks should be particularly careful [1]. However, due to disasters in land or sea traffic, accidental floods at fuel transshipment stations, as well as damage to transmission lines, these leaks pose a serious threat causing degradation of water and soil used for agriculture. In this

situation, the use of oil derivative sorbents is widely used [2–7]. In the case of oil derivatives, it is possible to use municipal solid waste composts or other cheap material, which makes it possible to reuse them as sorbents after the period necessary for biodegradation of absorbed compounds [8–10].

Sorbents which are designed for use on dry and wet surfaces (e.g., roads) should have a high sorption capacity under various temperature and humidity conditions.

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However, the sorption efficiency depends on the type of sorbent used (mainly the active surface, number and size of pores) of the adsorbate concentration, temperature, contact time of the adsorbate and adsorbent and mutual affinity of both these bodies [1]. Taking into account the need for soil and water resources protection, it is necessary to remove the organic liquid immediately and safely which is possible due to the application of sorbents whose main task is to retain the largest possible amount of hydrocarbons [2–4]. The currently used organic sorbents such as peat, sawdust, lignite, charcoal, sisal, bark, straw, nutshells, waste wool, coir fibers, cellulose, and various kinds of organic synthetics, are usually characterized by high sorption capacities and, in some cases, the capability to be reused multiple times during a single rescue operation [11,12]. Especially biochar was widely tested for the sorption of organic pollutants and also in case hydrophobic compounds and Persistent Organic Pollutants bound mechanisms were described [13]. In some cases (e.g., drugs removal from water solution) biomass fibers cannot be used due to very low sorption potential especially in front of activated carbon availability and good sorption properties of graphene oxide [14]. Moreover, mineral sorbents are used as well, especially in traffic accidents but usually, they are effective only for light non-aqueous phase liquid (LNAPL) layer removal. However, a used sorbent gains the properties of hazardous waste and has to be disposed of in landfills or utilized by incineration at specialized incineration plants. Many sorbents can be regenerated by removing, most often the breakdown of adsorbate particles, which makes it possible to reuse them, but in most cases, the used sorbent must be neutralized by burning or storage in a landfill for hazardous waste [10,15,16].

Synthetic fibrous materials used as sorbents are primarily products that are generated from polyvinyl chloride or polystyrene. Their sorption capacity is quite considerable and often exceeds 100 g of an oil-related product in proportion to 1 g of sorbent (over 1,000%). Furthermore, they can be neutralized in combustion plants for dangerous wastes, thus providing the opportunity to regain energy [17]. Still, other authors pay attention to another adsorption–desorption. This is the mechanical or chemical process of removal of the absorbed hydrocarbons through impressing or via a hexane rinse, for example, after which it is possible for the sorbent to be used again [18]. One way or another, the usage of sorbents that have been used previously to remove organic compounds dissolved in water is extraordinarily problematic. Road runoff produced from petrol stations and car parking areas is usually treated in separation tanks or evaporation ponds and removal of the LNAPL layer from the water surface is effective [19]. Aliphatic hydrocarbons (*n*-alkanes) are not so quite easily soluble in water but due to that fact, that in case of traffic accidents many other substances are released to the environment, for example, alcohols and detergents. In the final effect, *n*-alkanes solubility is much higher (concentrations can exceed 100 mg/dm<sup>3</sup>), but the process of sorption becomes more difficult [19,20]. What is more, the co-presence of surfactant character substances (coolant liquid or screen washer liquid) can additionally reduce the level of the compounds' activation on the sorbent's surface. Numerous studies have reported good sorption of contaminants on biomass-derived from different types of plants

[21,22]. The good direction in sorbent's material improvement is an easily available (for free or very cheap) biomass, including many species that are invasive in many countries (e.g., goldenrod), may be applied as cheap sorbents of oil-related substances [23,24]. *Typha latifolia* leaves biomass was used as an effective biosorbent of reactive dye from water, however, the preparation of sorbent was long and consumed a lot of energy [25]. Low cost of production, zero or low CO<sub>2</sub> emission and zero waste technologies are necessary to obtain circular economy and sustainable development.

The broadleaf cattail (*Typha latifolia* L.) seeds and coconut fiber suggested in this work are easy to get and environmentally friendly, full-biodegradable materials. *Typha* produces over 200,000 seeds per one flower and is available for free [26]. Therefore, they can be successfully used as cheap, easy to use hydrocarbons sorbent, that is available in different places all over the world.

*Typha latifolia* leaves and stems were tested as biosorbents of reactive dye or road runoff cleaning but using of seeds for *n*-alkanes sorption from a water solution has not been tested yet. Moreover in pre-tests relatively good results were obtained in LNAPL sorption from distilled water surface experiments. The aim of the research was to conduct a trial to test broadleaf cattail seeds (*Typha latifolia* L.), coconut fiber and peat fiber (as control ample) being applied for sorption aliphatic hydrocarbons dissolved/emulsified in water.

## 2. Materials and methods

### 2.1. *Typha* seed's fiber characteristics

The *Typha spadixes* that provided the *Typha* fiber-wool contain mature seeds in fresh autumn. Seeds of *Typha latifolia* looks like 1.8–2.0 cm diameter star with very thin fibers. The surface of fibers is hydrophobic (for long-distance water transportation), due to empty fiber tubes and a wax layer that cover the whole seed surface like the seeds of poplar or kapok plants [27]. In the case of this species, the main seed fiber usually is divided on various branched fibers. Each fiber is composed of a few long cells with septa and its surface is smooth with slight valleys between cellulose microfibers. The total length of *Typha* fiber is 6–10 mm (simple cellulose fiber has 250–875 μm) and in comparison with other weed seeds medium width – the main fiber has 45–50 μm and branches have 10–20 μm [26]. Plant fibers have many free hydroxyl groups at the molecular level that easily bond with oil or water, so a lot of types was used for petroleum products. Additionally, the mercerization process was introduced for sorption capacity enhance [17].

### 2.2. Material preparation

Broadleaf cattail dry *Typha spadixes* at the end of the vegetative cycle were collected in the Opole region (Poland). After harvest, the *Typha spadixes* were allowed to dry at open-air, in containers protected from the wind and after the expansion of seeds dried to constant mass at 80°C. Coconut fibers and peat sorbent were bought on the polish market and dried at 80°C prior to use. All used sorbents were mercerized in 5% NaOH.

The aliphatic hydrocarbons (*n*-alkanes C6–C15) sorption experiments were conducted as “batch-type” at a room temperature of  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ . The sorbents (Table 1) applied in the research were three types of natural materials: *B* – broadleaf cattail seeds (*Typha latifolia* L.), *C* – raw coconut (free fibers and chips) and *P* – commercial sorbent made of peat (Fig. 1). All used sorbents were mercerized. This process was conducted for 240 min. by soaking in hot ( $80^{\circ}\text{C}$ ) NaOH [28]. The hot water treatment (hydro-mercerization) process removed volatile compounds, waxy coatings, and extractives from cellulosic fibers, making them more accessible for the absorption medium but sodium hydroxide is more effective due to cellulose II production [24,29]. Prior to sorption experiments, all sorbents were dried again.

Total sorption capacity was tested with the procedure described by Wong et al. [17] but use 0.2 g of dried fiber soaked in  $20\text{ cm}^3$  of standard diesel oil available on market.

The testing of hydrocarbons sorption from the water solution was carried out in glass vessels (Erlenmeyer flasks) with a standard taper stopper. The water solution was prepared by dissolving hydrocarbons (Table 2) mixed in equal proportions (each  $0.5\text{ cm}^3$ ) hexane (C6), heptane (C7), ethane (C8), nonane (C9), decane (C10). The second

solution was made with undecane (C11), dodecane (C12), tridecane (C13), tetradecane (C14) and pentadecane (C15). Both solutions were made in  $1,000\text{ cm}^3$  of redistilled water containing methyl alcohol, ethylene glycol and screen washer liquid – each in the amount of  $5\text{ cm}^3/\text{dm}^3$ . The use of these additions was a simulation of different compounds (e.g., screen washer or engine coolant) which are present in regular road runoff effluent from a traffic accident, road runoff or sewage from the oil industry or petrol stations. It was a way for use of no-water soluble compounds (C11–C15) solution, where the main part of hydrocarbons was in form of a low-concentrated stable emulsion.

The obtained water solutions were kept at a temperature of  $20^{\circ}\text{C}$  in darkness. Prior to use, the undissolved hydrocarbons layer was removed from the water surface. Each time about 100 g of the dry sorbent were directly weighed out and placed into the Erlenmeyer flasks. The proportion of sorbent to water solution was 1:50 (m/v). After hydrocarbons solution addition, experimental flasks were vigorously shaken for 10 s.

The sorption time was 15, 30, 60, 90, 120, 240 and 360 min. After this time, the samples of the solution were taken and then submitted for extraction by dichloromethane.

Table 1  
Characteristic of sorbents used in experiments

Sorbent	Broadleaf ( <i>B</i> )	Coconut ( <i>C</i> )	Peat ( <i>P</i> )
Bulk density ( $\text{g}/\text{dm}^3$ )	1.666 (0.036)	72.42 (2.12)	92.8 (1.31)
Total sorption capacity ( $\text{g}/\text{g}$ )	22.47	3.52	6.04
Total pore area ( $\text{m}^2/\text{g}$ )	0.828	13.22	26.47
Mass of sorbed alkanes C6–C10 from solution $t = 30$ and $t = 360$ min ( $\text{mg}/\text{kg}$ )	209.5 220.1	309.8 357.8	340.1 360.0
Mass of sorbed alkanes C11–C15 from solution $t = 30$ and $t = 360$ min ( $\text{mg}/\text{kg}$ )	114.0 148.3	283.9 354.7	369.6 369.6
Sorption change C6–C10 (%) in $t = 30$ and $t = 360$	61.6/61.1	91.1/99.4	100/100
Sorption change C11–C15 (%) in $t = 30$ and $t = 360$	30.8/40.1	76.8/96.0	100/100



Fig. 1. Materials used in sorption experiments: *P* – peat; *C* – coconut; *B* – broadleaf before mercerization process (Photo: Ciesielczuk T).

Table 2  
Basic parameters of investigated compounds

Compound CAS number	Molar mass (g/mol)	Water solubility (20°C) (mg/dm <sup>3</sup> )	Density (g/cm <sup>3</sup> )
Hexane 110-54-3	86.18	12.5	0.6594
heptane 142-82-5	100.21	2.68	0.6838
Octane 111-65-9	114.23	0.66	0.7027
Nonane 111-84-2	128.20	0.122	0.7177
Decane 124-18-5	142.28	0.010	0.726
Undecane 1120-21-4	156.33	0.000	0.740
Dodecane 112-40-3	170.36	0.000	0.750
Tridecane 629-50-5	184.39	0.000	0.757
Tetradecane 629-59-4	198.42	0.000	0.760
Pentadecane 629-62-9	212.4	0.000	0.770

They were then shaken in laboratory vials (4 cm<sup>3</sup>) made of amber glass closed with a Teflon cap. The eluate was dried with Na<sub>2</sub>SO<sub>4</sub>, which had been previously stored at 130°C. All experiments were conducted in independent three replicate. Aliphatic hydrocarbons were analyzed in untreated extracts (or in blank samples) by the gas chromatography with flame ionization detector method, with the use of a capillary column VF-5MS (30 m; 0.25 mm ID; 0.25 µm film thickness), with a constant helium flow through the column in the amount of 1 cm<sup>3</sup>/min. The injector temperature was 260°C and the detectors were 280°C. The temperature program of the oven began at 90°C and was kept for 2.5 min; the temperature then rose at a rate of 7°C/min, up to 275°C. The final temperature of the program (275°C) was maintained for 3 min. The detection limit was 2.0–3.5 µg for a single compound. Two mathematical models were used to describe the kinetics—Lagergren's and Ho–McKay's [11].

### 3. Results and discussion

One of the more important parameters of the materials used in the process of sorption of petroleum substances in the content of the fibrous organic matter. The presence of a lot of fibrous made of cellulose in combination with lignin is a good base for the hydrocarbons and water sorption. In case tested materials, water sorption is very slow and especially raw peat and broadleaf seeds float over 24 h on the water table what have been observed in pre-tests. Based on earlier results, the mercerization process hot NaOH was the most effective (from all used mercerization procedures and chemicals) in sorption surface improvement, so it should improve sorption capacity [29]. After the mercerization procedure cellulose fibers changed the internal structure from cellulose I to alkali cellulose (Na-cellulose I) and then into cellulose II. In alkali cellulose distances between cellulose molecules are relatively big and other molecules (water) are bound and hydroxyl groups are changed in –O–Na structure. Intensive rinsing can remove Na ions and lead to obtain cellulose II – a stable form of this compound [28].

In general, the plant fibers exhibited a high affinity for oil and water but high hydrocarbons sorption is possible only in case of lack of water in the sorption medium

[17]. Total sorption capacity was the highest for broadleaf seeds (over 22 g/g) and much lower for coconut and peat (3.52 and 6.04 g/g, respectively) but it depends not only from the sorption-active material surface but mostly from chambers which are present between fibers of the sorbent. The total sorption capacity of peat sorbent tested in this work exceeded 6.0 g/g (it was lower than specified by the manufacturer – 8 g/g), but another part of this sorbent characterized only 4.2 g/g, so probably it depends from actual peat structure [10]. Similar *Typha* seeds total capacity were tested with SAE 10W-30 motor oil. Total efficiency of 26.39 g/g was obtained for untreated seeds that show small differences between procedures and seeds origin [31].

Two times higher amounts (40–50 g/g) of the absorbed oil-based products have been obtained when kapok fibers were applied (*Ceiba pentandra* L.) but the mean value of sorption obtained for natural fibers is low and usually between 1.7 and 3.7 g/g. Much higher amounts (nearly 100 and 85 g/g 24 h each) have been noted down when silk fibers which were used as a sorbent, but their usage is strictly dependent on the accessibility of wastes coming from textiles [32].

Obtained results of *n*-alkanes from water solution sorption, show very weak sorption properties in the case of broadleaf seeds what is opposition to the highest total sorption capacity and good results of LNAPL sorption in pre-tests. It shows a way for very cheap removal of oil/fuel leaks in petrol stations, car repair shops or even private garages. This, available for free material, has thick very light fibers and hydrophobic surfaces. Moreover, plant fibers have many free hydroxyl groups, which facilitate the attachment of hydrocarbons or water molecules [32]. The hydrocarbons water solution was a problematic medium for sorption for tested seeds. The removed wax layer in the mercerization process releases real cellulose surface and pores sorption capacity rise from 0.585 mm<sup>3</sup>/g in untreated seeds to 1.892 mm<sup>3</sup>/g in mercerized.

These properties were a good prognostic for the sorption of hydrocarbons from water solution. However rise of pore volume in mercerized material and structure of seed fibers is typical for plant material – like kapok or cotton and the expected total amount of adsorbed hydrocarbons should increase, obtained sorption results were weak.

Experiment solution properties was a cumulative effect of hydrocarbons water and used additions. So total sorption was a result of affinity of water, *n*-alkanes, alcohols and microcapsules made of hydrocarbons and nonionic surfactants (from screen washer liquid) to sorbent micropores surface.

In this case, after the end of the experiment time (360 min), dissolved hydrocarbons were still present in adsorbate (Fig. 2). Most important is the first 30 min. of

sorption and show real sorbent potential for use in real sorption conditions. Broadleaf sorbed over 209 mg/kg, but coconut over 309 mg/kg of C6–C10 alkanes (Fig. 3). The best results at 30 min, time was obtained for *P* – over 340 mg/kg (Fig. 4), which was much better than aromatic hydrocarbons (BTEX) sorption on broadleaf seeds [30]. In comparison to peat sorbent, the effectiveness of sorption was about 60%, but the coconut was much better – over 91% and 99% respectively. Sorption on broadleaf was especially weak in

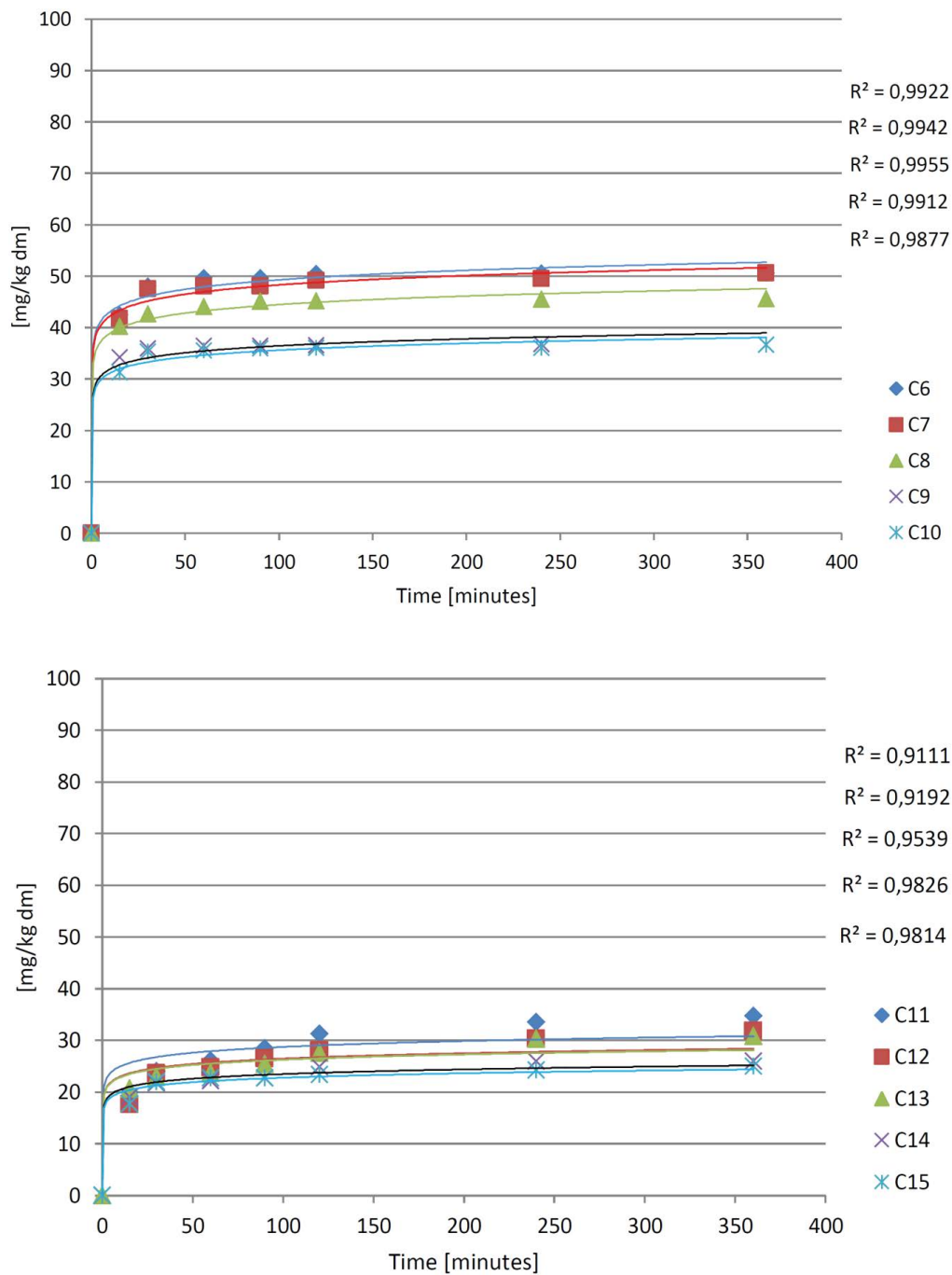


Fig. 2. *n*-alkanes sorption on mercerized broadleaf cattail seeds at 20°C.

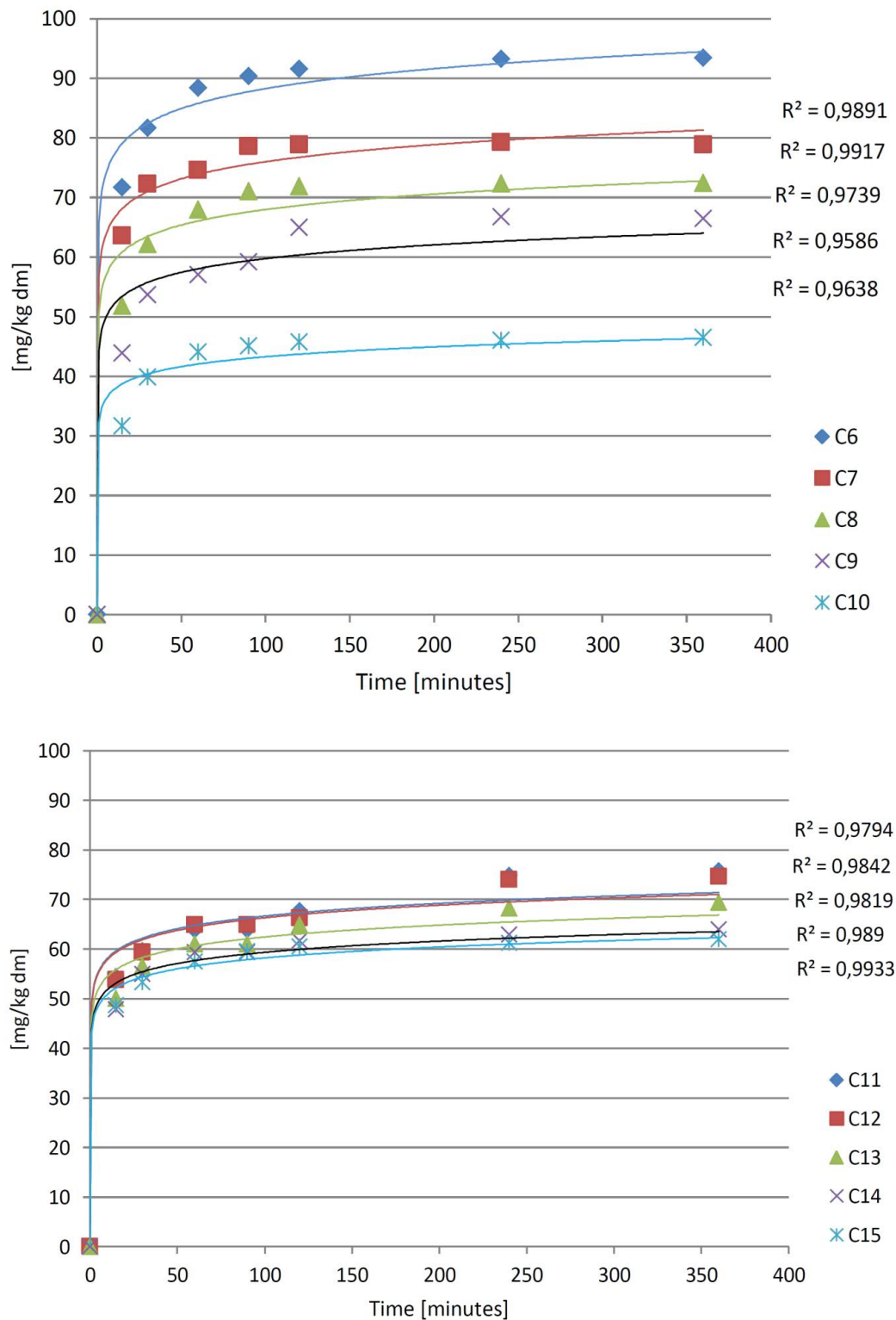


Fig. 3. *n*-alkanes sorption on mercerized coconut fiber at 20°C.

the second series with C11–C15 alkanes. Maximum sorbed hydrocarbons were 114.0 and 148.3 mg/kg at 30 and 360 min retention time. Respectively, what was only 30.8% and 40.1% in comparison to peat? In the case of coconut better

results were obtained – 76.8 and 96.0 for 30 and 360 min respectively. Due to this fact seeds appear as weak and coconut quite good sorption material. Sorption speed ( $k_1$  and  $k_2$  factors) calculated with Lagergren's and Ho–McKay's

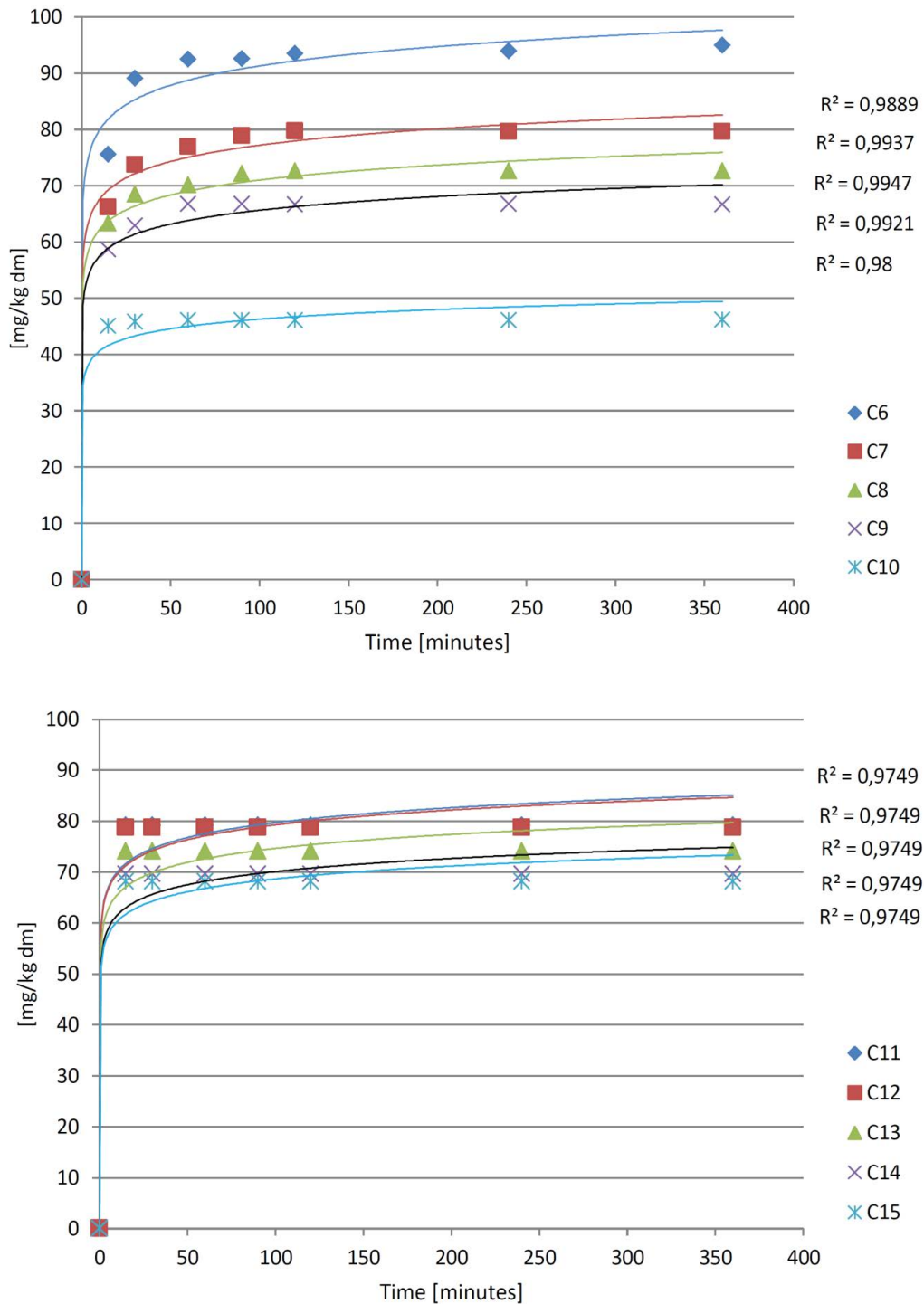


Fig. 4. *n*-alkanes sorption on mercerized peat fiber at 20°C.

models show a predominance of *P* material in the case of all analyzed compounds (Table 3) with high correlation factors (over 0.98) for pseudo-second-order equation.

Obtained results of experiments show great effectiveness of *B* in pure hydrocarbons liquid sorption, which can be useful in the case of petroleum products removal from a dry surface.

The optimal time for sorption is the first 30 min of sorbent action [5]. Prolongation of time in case *B* material does not lead to much better results – the speed of sorption is low and the total amount of hydrocarbons removed from solution slightly exceeds 220 and 148 mg/kg for C6–C10 and C11–C15 respectively. Obtained results from “batch type” experiments with cork and birch bark for *n*-alkanes



Table 3

Pseudo-first-order rate constant  $k_1$  and pseudo-second-order rate constant  $k_2$  for sorption of tested  $n$ -alkanes on analyzed sorbents

	$k_1$			$k_2$		
	<i>B</i>	<i>C</i>	<i>P</i>	<i>B</i>	<i>C</i>	<i>P</i>
C6	0.0073	0.0079	0.0091	2.31E-05	5.00E-06	8.33E-06
C7	0.0072	0.0075	0.0089	2.86E-05	1.10E-06	3.31E-06
C8	0.0065	0.0066	0.0083	6.68E-05	3.20E-05	5.25E-06
C9	0.0059	0.0061	0.0077	6.17E-04	3.20E-04	5.26E-05
C10	0.0043	0.0057	0.0073	5.14E-04	3.40E-04	2.58E-05
C11	0.0038	0.0050	0.0068	2.45E-04	8.60E-03	2.16E-05
C12	0.0032	0.0045	0.0062	1.56E-04	7.46E-03	9.42E-04
C13	0.0026	0.0039	0.0061	2.95E-03	2.00E-03	6.26E-04
C14	0.0020	0.0037	0.0059	5.46E-03	3.63E-03	4.11E-04
C15	0.0020	0.0037	0.0058	4.88E-03	9.17E-03	5.93E-03

removal were good – and varied between 90% and 100% of dissolved hydrocarbons were removed (like *C* material in this experiment) what confirm cheap sorbents effectiveness [20]. The problem of  $n$ -alkanes sorption with *B* material lies in hydrocarbons solution structure and seed surface structure. Low pore numbers were increased by mercerization but it was still ineffective. The sorption mechanism of hydrophobic hydrocarbons is based on hydrophobic interactions, pore-filling, H-bonding,  $\pi$ - $\pi$  electron donor-acceptor interactions and electrostatic attraction [13]. The problem of low sorption capacity of tested broadleaf seeds lies with adsorbate composition. In real conditions of traffic accidents and in our experiments, organic hydrophobic liquid (car fuel/hydrocarbons) is not real dissolved but suspended in form of ultrafine drops covered with surfactant molecules what in effect create a stable emulsion and dissolved light hydrocarbons, plus nonionic and ionic forms of Me-OH, nonionic ethylene glycol and not-bounded surfactant molecules. The stable emulsion contains ultrafine drops or even single molecules of  $n$ -alkane where hydrophobic groups of surfactant are connected to  $n$ -alkane drop, and hydrophilic to a water body. Because of the hydroxyl (-OH) groups in cellulose, these fibers usually have a high attraction for water and low concentrated solution of hydrocarbons covered by alcohols and nonionic surfactants from screen washer liquid have no possibility to find a free group and bound mechanisms as hydrophobic interactions, pore filling and  $\pi$ - $\pi$  electron interactions are present but with low intensity. These problems were not observed in experiments with monoaromatic hydrocarbons and only Me-OH was used as a “surfactant” factor [24]. Only high pore surface sorbents (materials *P* and *C*) have the possibility to remove high amounts of dissolved/emulsified hydrocarbons. Mercerization increases surface roughness and it increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites, but it is still not sufficient in the case of broadleaf seeds [28].

However real road runoff occurred in the form of a stable emulsion, conducted experiments show a different ability of dissolved hydrocarbons removal. Oil sorption by plant-origin fibers depends on many fiber-related factors. As the most important is the size, shape, structure/

arrangement (loose vs. mesh), fiber pretreatment, and the conditions in which fibers are exposed to the oil [17]. The best results were obtained for material *P* – commercial sorbent made of peat. This material was used as a control – very effective (high speed of sorption), high capacity natural material, designed for sorption of organic layer from water surface but peat is a non-renewable fossil material [10]. After intensive shaking surface of this material was moisture with water and sorption equilibrium was reached after 15 min – in the case of long-chain  $n$ -alkanes and about 60 min in the case of C6–C10 samples. In this case, water was purified in a very short time and no hydrocarbons were detected in adsorbate. Coconut fiber was found as a medium good material for sorption (due to differences in quality of market-available material), but obtained results were very good – close to very expensive peat. Equilibrium was reached after 120 and 240 min for short and long-chain alkanes respectively. So the main difference between peat and coconut was a time of equilibrium and of course the price of the sorbent. On the other hand, coconut fiber is a waste material (with low price) but expensive peat sorbent is a fossil material that will run out in about 50 y. All tested sorbents can be incinerated with energy recovery or composted after use, but it will increase CO<sub>2</sub> emission, so a better solution is composting with other biodegradable waste material.

#### 4. Conclusions

In the conducted laboratory experiments, sorption properties of mercerized broadleaf cattail (*Typha latifolia* L.) and coconut fiber have been verified in comparison to “high capacity” peat sorbent. In the course of these studies, it was shown that the amount of absorbed pollutions is dependent on the contact time of the polluted water and a sorbent type. Weak results were obtained in experiments with broadleaf seeds and the best for commercial sorbent made of peat. Coconut fiber – a waste material, appears as the best for sorption  $n$ -alkanes from solution due to good sorption properties and low price. However broadleaf seeds have a hydrophobic surface, thick fibers and have the highest total hydrocarbons capacity and can be used for free hydrocarbons sorption (e.g., LNAPL, or petrol stations or garage oil/



fuel leaks), but cannot be used as sorbents of dissolved or/and emulsified *n*-alkanes with good results. The significant hydrophobicity of the analyzed materials makes the contact between the seed's surface and an adsorbate more difficult, thus extending the time for the process.

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