



## Application of the water-insoluble pyrolysis oil fraction as an organic binder



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### HIGHLIGHTS

- The water-insoluble fraction of pyrolysis oil can be used as a component of bitumen binder.
- The best properties are exhibited by a composite binder supplemented with 10–15% wiPO.
- A composite binder with an adhesive strength higher than that of both initial materials is obtained.
- Modified asphalt concrete has lower water absorption and higher strength.

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### ABSTRACT

Wood fast pyrolysis oil could potentially be applied as an additive to an organic binder for use in road construction. The aim of this work was to investigate the properties of a composite binder made of bitumen and the water-insoluble fraction of wood pyrolysis oil. Pyrolysis oil was made from birch wood by fast ablative pyrolysis. Separation of the pyrolysis oil was conducted with recovery of the water-insoluble fraction of wood pyrolysis oil. A composite binder consisting of bitumen and the water-insoluble fraction of pyrolysis oil was produced. Modified asphalt concrete was made with the composite binder. The properties of the composite binder and the modified asphalt concrete were investigated. It can be concluded that the water-insoluble fraction of pyrolysis oil can be used as a component of composite bitumen binder. The best properties are exhibited by the binder supplemented with 10–15% of the water-insoluble fraction of pyrolysis oil.

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## 1. Introduction

Most bituminous adhesives or binders used for pavement materials are derived primarily from fossil fuels [1]. However, the reduction of fossil feedstock reserves has resulted in the desire to involve renewable feedstock in industry. Thus, the use of renewable resources and materials, including in road construction, is a significant area.

Lignocellulosic biomass is one of the largest and most widely available renewable resources on Earth [2]. Biomass processing in wood industry, agriculture and other branches results in the generation of large quantities of wastes. These wastes often remain unclaimed and require money and effort for utilization. However,

this renewable resource can be used as feedstock for the production of substitutes for fossil materials. Fast pyrolysis is one of the promising technologies to convert biomass into liquid products, which can be used as a substitute for petroleum-derived products in different applications [3].

In addition, liquid products of the thermal decomposition of lignocellulosic biomass may be applied as additives to an organic binder in road construction [4]. The application of wood thermal decomposition products in road binders and for soil strengthening has been represented in several works [5–7]. This approach is particularly relevant when involving forestry wastes and the use of local renewable materials as a binder in road construction.

The highest yield of liquid products (up to 75 wt.% [8]) can be achieved by fast pyrolysis, i.e., thermal decomposition of biopolymers in the absence of an oxidizing environment at high (up to 1000 °C/s) heating rates and low product residence times in the reaction zone [9]. Liquid products of fast pyrolysis include a

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plurality of compounds with different properties resulting from the thermal decomposition of the main biopolymers of cellulose and lignin [10]. In particular, they include both water-soluble (low-molecular-weight carboxylic acids, ketones, aldehydes, hydroxyacetaldehydes, anhydrosugars, “sugars”, etc.) and water-insoluble components, also known as “pyrolytic lignin” (resins, lignin polymers and oligomers, aromatics, etc.) [11]. Compounds and polymers that are not exposed to leaching and dissolution in water are of greater potential interest for applications in road binders. Functional hydroxyl, methoxyl, carbonyl and carboxyl groups contained in these compounds can potentially increase the adhesion strength of the binder to mineral materials by physical and chemical interaction with major groups of carbonate rocks of the mineral part of asphalt concrete and can also slow down the process of thermal oxidative degradation of bitumen [12].

Research studies on the application of fast pyrolysis liquid products and their fractions in road construction have been conducted by a number of scientists at the University of Iowa (USA), as well as specialists of BTG Company (the Netherlands) [13,14]. The results showed promising prospects for their use as a binder modifier.

Thus, the application of the water-insoluble fraction of pyrolysis oil as a binder modifier is a very interesting research area. The purpose of this work was to investigate the influence of the addition of water-insoluble pyrolysis oil on binder properties.

## 2. Materials and methods

### 2.1. Production of pyrolysis oil

Fast pyrolysis oil was produced on a pilot plant [15] in ablative mode from grinded birch wood at the temperature  $500 \pm 20$  °C. The initial moisture of the wood was  $8 \pm 0.5\%$ . Condensation of pyrolysis oil was conducted at  $30\text{--}40$  °C. The grain size of the feedstock was 0.5–5 mm. Pyrolysis oil has a dark brown or black color and a smoky odor.

### 2.2. Properties of pyrolysis oil

The primary physical properties of the pyrolysis oil are presented in Table 1.

The density of the pyrolysis oil was identified with an AON-1 985 hydrometer via the standard method [16] with an accuracy of  $\pm 0.5$  kg/m<sup>3</sup>. The higher heating value of the pyrolysis oil was determined with a IKA C 5000 calorimeter. The measurement was conducted in isoperibolic mode on a 0.5-g sample with excess oxygen at a temperature of 25 °C [17]. The pH indicator was measured with a Mark-901 pH meter and ESK-10601/7 combined glass electrode. The water content was investigated with a V-20 automatic Karl Fischer titrator for volumetric titration, Metler-Toledo, by the standard method [18]. Measurement of the viscosity of the pyrolysis oil was conducted with a VZ-246 flow-cup viscometer, for which the diameter of the nozzle was 6 mm. The measurement was conducted at  $20 \pm 2$  °C. The results in seconds were converted to mm<sup>2</sup>/s by the standard method [19].

### 2.3. Preparation and properties of the components of the composite binder

The composite binder utilized for testing was made of bitumen and the water-insoluble part of pyrolysis oil. Bitumen properties are presented in Table 2.

Preparation of the water-insoluble fraction of pyrolysis oil (wiPO) was carried out by water extraction and sedimentation of the water-insoluble fraction. A mixture of pyrolysis oil and water in a ratio of 1:10 was placed in an ultrasonic bath for 30 min. Then, the mixture was placed in a centrifuge for 30 min. The rotation frequency was 4500 min<sup>-1</sup>. After sedimentation, the water-soluble fraction was merged. The residue in the centrifuge tube was washed by distilled water one more time. The water content of wiPO was 38.5%.

**Table 1**  
Physical properties of pyrolysis oil.

Property	Value
Density	$1200 \pm 1.0$ kg/m <sup>3</sup>
Higher heating value	$20.2 \pm 1.6$ MJ/kg
pH	$2.82 \pm 0.3$
Water content	$17.18 \pm 1.05\%$
Viscosity	$2.32 \pm 0.32$ mm <sup>2</sup> /s

**Table 2**  
Physical properties of bitumen.

Property	Value
Penetration at 25 °C	$6.15 \pm 0.5$ mm
Softening temperature	$57 \pm 1$ °C
Tensile at 25 °C	$49 \pm 0.8$ cm
Fraas brittleness	$-22 \pm 0.3$ °C

For production of the composite binder for road construction, bitumen was heated to 120 °C. The water-insoluble fraction of pyrolysis oil was heated to 80–90 °C for 30 min and then mixed with the bitumen in an amount from 0 to 100%. After heating at 80–90 °C for 30 min, the water content of wiPO was 11.8%.

### 2.4. Properties of the composite binder

For use of pyrolysis oil as a binder component, the properties of the binders were determined. Before the test, the binder sample was dehydrated by a gentle heating to 120 °C without overheating. The dewatered and melted composite binder was passed through a sieve and then thoroughly stirred to completely remove the air bubbles. The mass of the binder sample was approximately 50 g.

The softening temperature was determined by the ring and ball method according to ASTM D36 [20]. Two horizontal disks of the binder were cast in shouldered brass rings and heated with a controlled rate in a liquid bath while each of the disks supported a steel ball. The softening point was reported as the mean of the temperatures at which the two disks softened enough to allow each ball, enveloped in binder, to fall a distance of 25 mm.

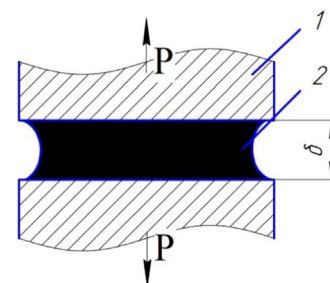
Determination of binder tensile was carried out by the standard method of ASTM D6084 [21]. Molten binder was poured into three forms by a thin stream from one end of the form to another until it was filled up to the edges. Binder potted in the form was cooled in air for 30–40 min at  $25 \pm 5$  °C, and then the excess binder was smoothly cut off by a hot knife from the middle to the edges so that the binder fills out a form up to the edges. Forms with binder, without being removed from the plate, were placed in a bath of ductilometer. The height of the water layer above the binder was 25 mm, and the bath temperature was maintained at  $25 \pm 0.5$  °C. After 1 h, the forms with binder were removed from the bath, taken from the plate and mounted in the ductilometer. After that, side parts of the forms were removed. The tensile speed during the test was 5 cm/min. Binder extensibility was measured as a length of binder string marked by the pointer at the point of rupture. For each sample, three definitions were conducted. For the final result, the arithmetic mean value of the triplicate determinations was accepted.

The Fraas brittleness of the binder was determined by the standard method according to EN 12593 [22]. A sample of binder was applied to a metal plate at an even thickness. This plate was subjected to a constant cooling rate and flexed repeatedly until the binder layer broke. The temperature at which the first crack appeared was reported as the Fraas breaking point.

The standard method according to ASTM 5-97 [23] was used to determine the depth of needle penetration. A sample of binder was melted and cooled in air at 25 °C for 90 min. The penetration was measured with a penetrometer by means of which a standard needle was applied to the sample at 25 °C.

The adhesion strength on the border between binder and stone material was determined by the Kisilyov method [6] of direct measurement, the essence of which consists of tearing marble plates glued together with a thin layer of the composite binder from each other (Fig. 1). The surface of the polished marble plates was previously well cleaned, dehydrated and degreased with solvents: ethanol and then diethyl ether.

A sample of composite binder was melted at 80–90 °C and then placed between the polished surfaces of horizontally disposed marble plates. The upper plate was pressed against the bottom with a load of 3 kgf/cm<sup>2</sup>. Within 15 min, the



1 – marble plate; 2 – layer of binder

**Fig. 1.** Scheme of the tearing of the composite binder by marble plates.

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