

## Determination of Mass of Bitumen in Bitumen Sheets by Calcination Method

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### Abstract

The work analyses the problem of determination of the mass of bitumen in waterproofing membranes - bitumen sheets by method of the calcination. The waterproofing function in bitumen sheets is ensured by the bitumen mass. The amount of bitumen mass is not prescribed in the European standards for bitumen sheets. The amount of bitumen is specified in some national standards. The amount of bitumen was determined by the extraction method and the calcination method for bitumen sheets and the sample of bitumen coating. The calcination method is not described in the standards, only in the forthcoming standard prEN 544:2020 for bitumen sheets. To compare the two methods, only the sample of bitumen sheet and a sample of bitumen coating material were used. The coating of bitumen sheet and the sample of bitumen mass were elastomeric character (SBS). The bitumen coating of known composition was prepared in the laboratory. By comparing results of both methods, it was found that the mass of the bitumen obtained from the bitumen coating material by extraction was 0.7 % higher than the mass of the bitumen obtained by the calcination method. This small difference was due to the fact that the same amount of bitumen coating was tested. During the testing of the bitumen sheet, it was found that the calcination time of 2 hours according to prEN 544:2020 is insufficient for the calcination of bitumen sheets. The mass of the bitumen obtained from the bitumen sheets by extraction was 0.6 - 1.3% lower than the mass of bitumen obtained by the calcination method. The termination of the calcination was performed when the weight loss of the test specimen was less than 0.03 g. The time of the calcination of the bitumen sheet and the bitumen coating material, with a comparable amount of bitumen material, differed by 0.5 hour, for the calcination of bitumen sheets at least 4 hours. The prEN 544:2020 methodology cannot be used. The difference in the results of both methods was 0.6 - 1.3 % depending on the considered mass of bitumen of the carrier. The higher difference between the two methods is due to the different thickness and the mass of bitumen of the test specimens. In contrast to the bitumen coating material, the mass of bitumen of the bitumen sheet differed for both methods. Furthermore, it was found that the weight and arrangement of the test specimen in the incinerating dish has an effect on the time of the calcination. The calcination method is as the time consuming as the extraction method for bitumen sheets with the bitumen coating material of elastomeric character. The extraction method is more accurate.

**Keywords:** Waterproofing membrane; bitumen sheets; calcination; extraction; mass of bitumen; bitumen coating.

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### 1. Introduction to the issue - why determine the mass of bitumen in bitumen sheets, is this the only way?

This work deals with the issue of determining the mass of bitumen in waterproofing membranes - bitumen sheets and is directly related to the article [1]. Bitumen sheets in the construction industry primarily fulfil a waterproofing function. The waterproofing function in bitumen sheets is provided by bitumen [2]. Bitumen sheets are produced according to European product standards. The requirement for the mass of bitumen in bitumen sheets is not specified in these standards.

In the case of the Czech Republic, it is ČSN 730605-1 [3], which determines the mass of bitumen in bitumen sheets, which are produced according to standards EN 13707 [4], EN

13969 [5] EN 13 970 [6]. The test procedure for the determination of the mass of bitumen in bitumen sheets in the case of the Czech Republic is given directly in ČSN 730605-1, Annex D [3].

The mass of bitumen is determined here by Soxhlet extraction. This issue is analysed in more detail in [1]. The extraction is a separation process in which the substances contained in one phase of the material are divided into other phases on the basis of different solubilities in the used solvent.

In practice, however, it is possible to determine the mass of bitumen in bitumen sheets by two methods, namely the extraction or the calcination. According to the information available to us (to the period of 9/2021), the calcination method has not yet been described in any of the test regulations, which deals with the determination of the mass of bitumen in bitumen sheets (this topic was analysed only in [7]). The method of the calcination can be included to thermal analyses, which are the most commonly used. The principle of thermal analysis is based on the removal of combustible parts from bitumen sheets. The calcination method is dealt only with in the forthcoming standard on bitumen shingles prEN 544:2020 [8]. The authors participated in commenting on this standard within CEN 250 thanks to the cooperation with the Association of Manufacturers of Bitumen sheets in the Czech Republic. prEN 544:2020 [8] can be the starting point for the development of the separate methodology that determines the mass of bitumen in bitumen sheets.

The aim of this work is to compare the accuracy of obtained results by the extraction method and the calcination method. The comparison will be made on a sample of bitumen sheets and a sample of bitumen coating. The sample of the bitumen coating material will be prepared in the laboratory, so the exact amount of bitumen and fillers will be known. This sample of known composition will be used to compare the results of individual methods. The weight of the bitumen coating test specimen corresponds to the weight of bitumen coating mass in the bitumen sheets test specimen. By the calcination of the test specimen of the bitumen coating material with a known weight of the bitumen material, we find out the necessary time for the calcination. Three basic hypotheses were established:

**The first hypothesis:** The weight, size and arrangement of the test specimen in the incinerating dish affect the time of the calcination.

**The second hypothesis:** It will not be possible to use the test procedure according to P EN 544 [8] for bitumen sheets. The calcination time of 2 hours according to prEN 544 [8] will not be sufficient for bitumen sheets.

**The third hypothesis:** Differences between results of individual methods (the extraction and the calcination) for the sample of bitumen sheet and bitumen coating material will be up to 2 %. The 2 % limit was chosen for the following reason. The amount of bitumen for bitumen sheets 4.2 mm thick which are used for roof waterproofing according to [ČSN 730605-1] [3] is 2 500 g/m<sup>2</sup>. According to our experience, the usual mass of bitumen of the PES carrier for bitumen sheets is 150 - 250 g/m<sup>2</sup> according to [9-11], so 200 g/m<sup>2</sup> ±50 g/m<sup>2</sup> and just 50 g represents 2 %.

## 2. Material and methodology

### 2.1. Methodology - test procedure

The work can be divided into five sections:

1. section - the preparation of bitumen coating material in the laboratory,
2. section - the calcination of bitumen coating material,
3. section - the test calcination of the bitumen sheet,
4. section - the calcination of the bitumen sheet,
5. section - the extraction of the bitumen sheet and the sample of bitumen coating material.

#### 2.1.1. Preparation of bitumen coating material in the laboratory

Bitumen coating material weighing approximately 650 g was prepared in the laboratory. Three samples weighing 5 g, 10 g and 15 g were prepared from this material. Due to the possible sedimentation of fillers in the bitumen mass, one test specimen from one sample was

always prepared. Another test specimen were prepared after mixing the bitumen mass. Each sample contained a set of three test specimens. Test specimens were weighed with the precision of 0.01 g.

To determine the time of the calcination of the maximal amount of the bitumen mass the sample weighing 20 g has been prepared. The weight of the bitumen coating material - 20 g corresponds to the assumed maximal amount of bitumen mass in the test specimen measuring 50 x 100 mm. The weight of the bitumen mass is determined in [3]. The bitumen sheet for single-layer applications 5.0 mm thick according to [3] contains at least 2 900 g/m<sup>2</sup> of the bitumen. Assuming at least 35 % of the amount of fillers [9-10] is the mass of the test specimen measuring 100 x 100 mm is equal to the value 39.15 = 29 x 1.35 g. In one incinerating dish there was placed the test specimen with an area of 50 x 100 mm. The mass of the test specimen was 39.15 g.

The bitumen sheet 4.2 mm thick contains the minimal amount of bitumen mass in the value of 2 500 g/m<sup>2</sup> according to [3]. Assuming at least 25 % of the amount of fillers [9-10] the mass of a test specimen measuring 100 x 100 mm is equal to the value at least 31.5 g = 25 x 1.25. In one incinerating dish there has been placed the test specimen with an area of 50 x 100 mm. The mass of the test specimen was 31.5 g.

For research purposes, the weight of the bitumen of the coating mass of the test specimen measuring 100 x 100 mm was rounded to 40 g and 30 g. One incinerating dish obtained a test specimen measuring 50 x 100 mm. The mass of the bitumen of the test specimen was thus set at half due to the floor area of the furnace.

It was necessary to use the sample of 20 g to determine the calcination time. The calcination time for the sample of 20 g has been determined using a regression function according to time for 5 g, 10 g and 15 g of bitumen coating material. The expected calcination time of the test specimen of the bitumen sheet was in the interval of the calcination time of 15 g - 20 g of bitumen coating material.

### **2.1.2. The calcination of the bitumen coating material**

According standard [8], the test was performed on three test specimens measuring 50 x 100 mm, the calcination temperature was 600 °C, the weighting accuracy was 0.01 g and the time of calcination was 2 hours. The tempering of the test specimen and the calcination dish are not specified. The calcination of bitumen coating was finished when the weight loss was less than 0.03 g, see [1]. The weight loss was monitored in a 0.5 hour interval with the first measurement after 1 hour. The resulting time to end the calcination has been calculated as the arithmetic mean of the times from the measurements of three test specimens.

### **2.1.3. Test calcination of the bitumen sheet**

In this stage, the comparison of the weight, size and arrangement of the test specimens of the bitumen sheets in the incinerating dishes has been solved. It was possible to use combustion crucibles or dishes for the calcination. Incinerating dishes have been used for the test. For economic reasons, porcelain incinerating (laboratory porcelain) dishes have been chosen. The comparison of the arrangement of the test specimen in the incinerating dish was based on:

- the size of commonly available incinerating ceramic dishes,
- the indoor floor plan of the muffle laboratory furnace,
- the time required to prepare the test specimen.

The test should be performed as soon as possible. All three test specimens of test pieces should be tested at the same time and the test specimens should be ready for incinerating dishes as soon as possible. There are two suitable sizes of dishes – the low form of the height of 20 mm with the diameter of 81 mm, the deep form of the height of 35 mm and with the diameter of 70 mm, see Fig. 1. The size of the floor plan of the furnace is 210 x 255 mm. The maximal number of incinerating dishes placed in this location was six. One test specimen measuring 100 x 100 mm was therefore divided into two incinerating dishes. In a low dish, marked V04, three layers of bitumen sheets were placed - two layers measuring 25 x 50 mm and one measuring 50 x 50 mm. Four 25 x 50 mm layers were placed in the deep dish, labelled V02.

For clarification a test sample size of 50 x 50 mm was tested for dishes of both sizes marked as test specimens V01, V03. Variants of the arrangement of test specimens are shown in Fig. 2. The selected incinerating dish of the arrangement could be used for further tests of calcination of bitumen sheets.

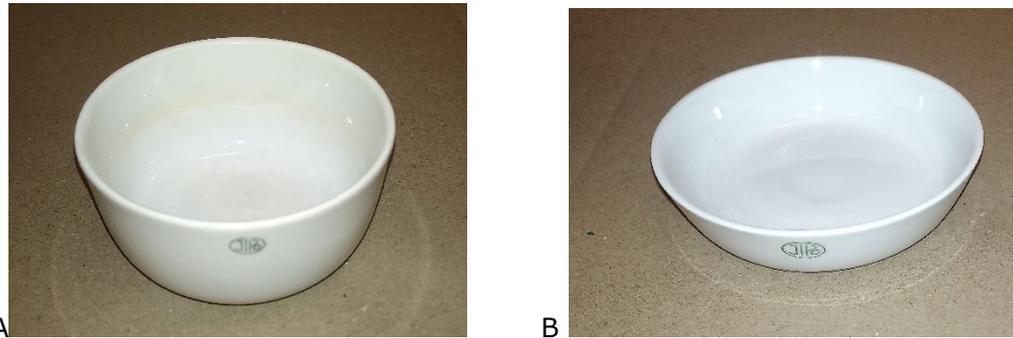


Figure 1. Calcination dishes. A – Incinerating dish, deep form, B – Incinerating dish, low form

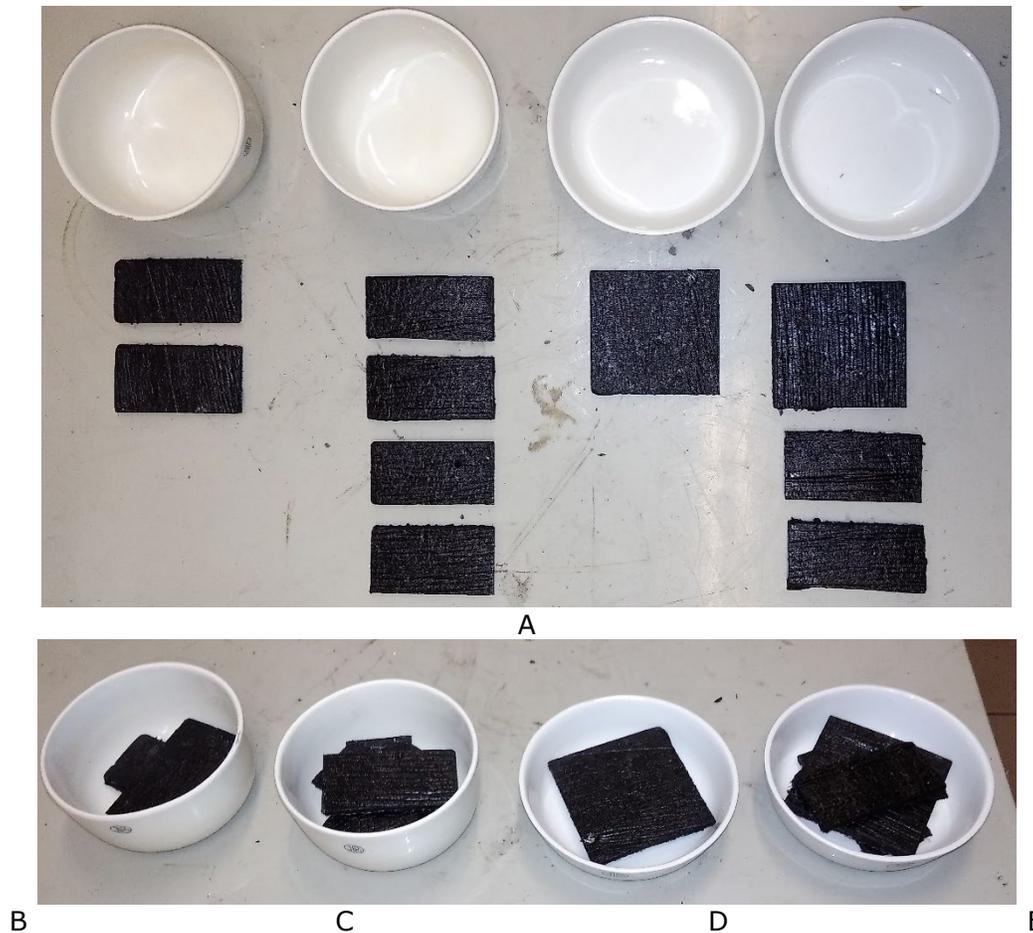


Figure 2. The location of test specimens in the dish. A - dish with test specimens, B – V01 incinerating dish, deep form, 2 layers, C – V02 incinerating dish, deep form, 4 layers, D – V03 incinerating dish. Low form, 1 layer, E – V04 incinerating dish, low form, 3 layers

#### 2.1.4. Calcination of bitumen sheet

The test procedure for the calcination has been based on the prepared standard [8]. In accordance with [1,8], three test specimens were used. The size of the test specimen was

increased from 50 x 100 mm [8] to 100 mm x 100 mm [1] due to the comparison of results between two methods (calcination, extraction). The test specimens were taken from the edges and the centre of the bitumen sheets sample, with the end test specimen being 100 mm from the edge of the bitumen sheets [1]. Test specimens for calcination were selected from the same sample as for extraction, for the location of test specimens see Fig. 3, 4. The temperature of the calcination was in accordance with [8], namely 600°C. The time of the calcination was verified by the calcination until the weight loss was less than 0.03 g in intervals 0.5 hour, see [1]. The resulting time of the calcination has been calculated as the arithmetic mean of times from measurements of three test specimens.

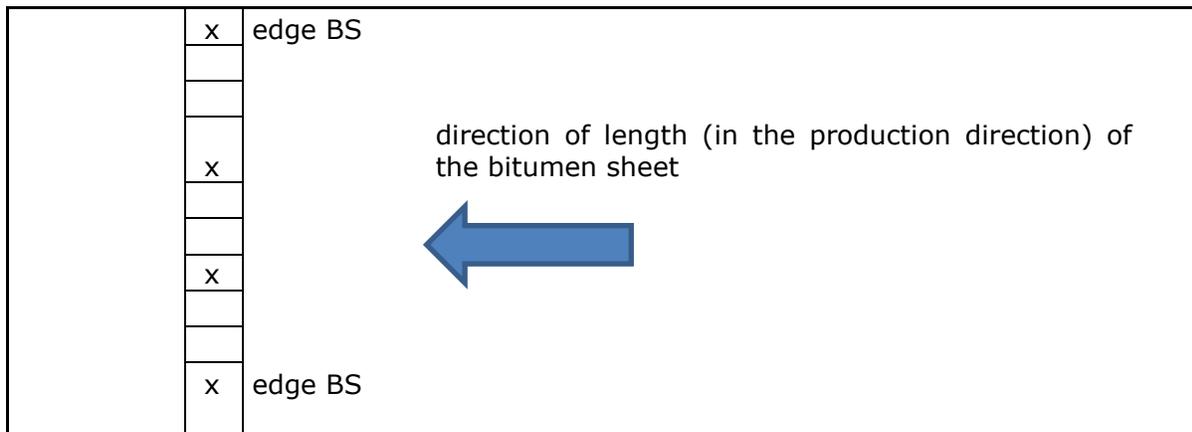


Figure 3. The sample of bitumen sheet with the indication of the location of test pieces. Source: Own.

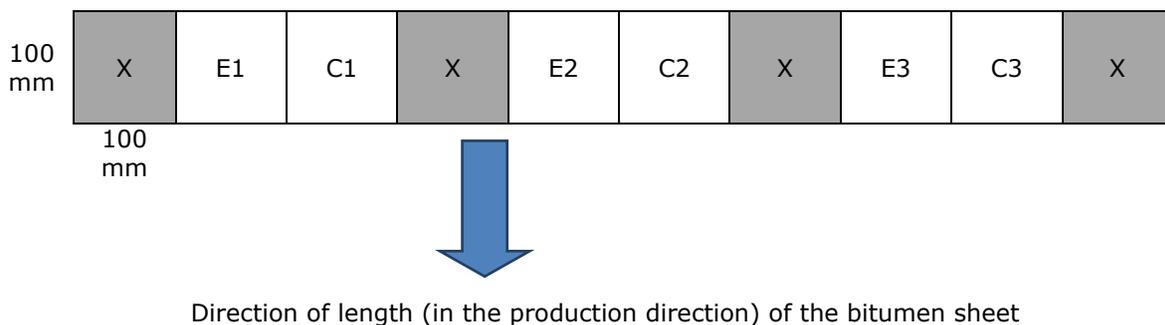


Figure 4. The location of test specimens in the test piece of bitumen sheet.  
E1, E2, E3 – test specimens for extraction, C1, C2, C3 – test specimens for the calcination

To determine the amount of bitumen mass in the bitumen sheet, it is necessary to subtract other combustible residues from the obtained mass of bitumen. If the bitumen sheet contains a flammable polyester carrier, the average usual mass of bitumen of the carrier is 200 g/m<sup>2</sup> [9- 11]. The actual mass of bitumen of the carrier can be verified by extraction. Other combustible residues can be separating foils on the upper or lower surface of the bitumen sheet. The mass of bitumen of the separating and protective polymer film is usually 20 g/m<sup>2</sup> [9, 12].

**2.1.5. Extraction of bitumen sheet and sample of bitumen coating material**

The test procedure for extraction has been based on [1]. The test procedure was maintained with the test specimens placed in the extractor folded into the filter paper. Preparation and selection of test specimens for both methods (extraction, calcination) was performed according to [1]. The test specimen weight of the bitumen coating sample has been set at 15 g. The test specimen had the same weight as zhe test specimen for both the calcination and the extraction.

## 2.2. Materials – bitumen sheet and sample of bitumen coating material

The bitumen sheet was selected for the tests of bitumen in bitumen sheets, which is used as the final layer in waterproofing roofs with a material treated with polymers of elastomeric character. The sample of bitumen coating material contained bitumen material of elastomeric character. The sample of bitumen coating material weighing approximately 650 g was prepared in the laboratory. The sample of primary bitumen 160/220 according to ČSN EN 12591: 2009 [13] was treated with 8 % polymers of elastomeric type SBS. It was a type of Kraton 1184 in powder form. The sample of bitumen coating material (recipe) contains 70 % of bitumen material (bitumen + polymer) and 30 % of fillers. Polymers of the elastomeric type have been chosen because this type of modification is most often used in the conditions of the Czech Republic, see [1,9-10,14]. The used primary bitumen was produced from URAL crude oil by vacuum distillation. Concrete fly ash was used as a filler ČSN EN 450-1 [15]. Specifications of bitumen sheet and sample of bitumen coating material are described in Table 1.

Table 1. Overview of used samples

| Monitored properties/signification     | Bitumen sheet SBS  | Sample of bitumen coating material   |
|--|--|--------------------------------------|
| thickness mm/<br>top surface treatment | 4.2 mm<br>coarse-grained mineral grit<br>easily combustible foil | -<br>without sprinkles               |
| bottom surface treatment               | PE + PP  | without modification                 |
| bitumen mass modification<br>carrier   | elastomeric<br>polyester   | elastomeric<br>without reinforcement |
| mass of the carrier                    | 160 - 200 g/m <sup>2</sup>                                       | -                                    |

## 2.3. Extraction materials - solvents

Based on previous research [1], xylene was used as the solvent.

## 2.4. Testing equipment - aids and devices

The following aids and materials were used for the tests: for the extraction of the device according to [1,3], the extraction device according to Soxhlet with an extension with a volume of 500 mL, the heating nest with a volume of 2 000 mL, the weighing-machine with an accuracy of 0.01 g, the dryer with forced air circulation, the muffle laboratory furnace, the mixer for the preparation of bitumen, oil bath and protective equipment.

## 2.5. Evaluation methodology

### 2.5.1. The preparation of bitumen coating material in the laboratory

The bitumen mass was sufficiently mixed for the time of 30 minutes, at 170°C and 800 rpm and showed an elastomeric character.

### 2.5.2. The calcination of bitumen coating material

The time of the calcination and the amount of bitumen has been verified. With less bitumen, the calcination time would be shortened. The amount of bitumen mass in the bitumen coating mass was compared with the amount known from the recipe. In the case of bitumen coating, it is assumed that the amount of bitumen is at least the same (70 %) or higher than in the recipe. Our practical experience shows that for small mixtures (up to 1000 mL) the results differ by up to about 2 %. The reason is the fillers and part of the polymer. Some of the fillers and polymer remain on the walls of the mixing vessel and on the stirrer. The resulting mass of bitumen should be reach of values between 70 – 72 %. If the difference is greater, then the new bitumen coating material would be prepared.

### 2.5.3. The test calcination of the bitumen sheet

The selection criterions for the incinerating dish are:

- the mass must remain in the calcination dish,
- the shortest possible calcination time,
- good handling of the calcination dish in laboratory tongs.

#### 2.5.4. The calcination of the bitumen sheet

The sample of bitumen sheets was calcined. The expected calcination time of the test specimen of bitumen sheets were in the range of the calcination time of 15 g - 20 g of bitumen coating material.

#### 2.5.5. The extraction of the bitumen sheet and sample of the bitumen coating material

The results obtained by the extraction have been compared with the results obtained by the calcination. The results related to the bitumen coating sample should be more accurate than the results related to the bitumen sheets. In the case of bitumen coating, this is a possible error in the preparation of the bitumen coating. In the case of bitumen sheets, this is an error which is given by the different mass of the test specimens and the estimate of other combustible samples (weights of the carrier, PE foil).

If the differences between the results of the individual methods (extraction and calcination) are up to 2 %, the result can be considered excellent. If the value of the result is higher, it will be necessary to adjust the weight loss limit of the bitumen mass to be 0.03 g, to review the mass of the bitumen carrier, to recalculate the results to the same mass of the bitumen sheet test specimen. It is considered that the extraction time of 24 h according to [1] is sufficient.

### 3. Results

#### 3.1. The calcination of the bitumen coating material

Table 2. Overview of the results of the calcination of the sample of the bitumen coating material

| Test piece | Test specimen                            | Weight of dish<br>[g] | Weight of bitumen coating mass<br>[g] | Weight of sheet with dish<br>[g] | Calcination weight of non-combustible residue with dish is finished when the increase is less than<br>0.01 - 0.03 g |       |       |       |       |       |       |
|------------|--|-----------------------|---------------------------------------|----------------------------------|---|-------|-------|-------|-------|-------|-------|
|            |  |                       |                                       |                                  | 1 h   | 2 h   | 2.5 h | 3 h   | 3.5 h | 4 h   | 4,5 h |
| V15        | V151                                     | 51.85                 | 15.1                                  | 66.83                            | 57.83   | 57.18 | 56.77 | 56.33 | 56.02 | 56.01 | 56.04 |
|            | V152                                     | 53.48                 | 15.00                                 | 68.42                            | 59.42   | 58.86 | 58.54 | 58.22 | 57.89 | 57.65 | 57.64 |
|            | V152                                     | 53.06                 | 15.1                                  | 68.04                            | 59.03   | 58.28 | 57.81 | 57.39 | 57.23 | 57.21 | 57.21 |
|            | amount of bitumen in bitumen coating [%] |                       |                                       |                                  | 60.0  | 64.3  | 67.0  | 69.6  | 71.4  | 72.0  | 72.0  |
| V10        | V101                                     | 58.52                 | 10.00                                 | 68.52                            | 62.42   | 61.65 | 61.24 | 61.20 | 61.23 | -     | -     |
|            | V102                                     | 49.17                 | 10.00                                 | 59.10                            | 52.97   | 52.10 | 51.79 | 51.79 | 51.82 | -     | -     |
|            | V103                                     | 55.88                 | 10.00                                 | 65.86                            | 59.77   | 59.01 | 58.59 | 58.61 | 58.61 | -     | -     |
|            | amount of bitumen in bitumen coating [%] |                       |                                       |                                  | 61.1  | 69.1  | 72.9  | 72.9  | 72.7  | -     | -     |
| V05        | V051                                     | 52.3                  | 5.00                                  | 57.39                            | 54.23   | 53.69 | 53.65 | 53.64 | 53.64 | -     | -     |
|            | V052                                     | 59.84                 | 5.00                                  | 64.94                            | 61.71   | 61.20 | 61.14 | 61.15 | 61.15 | -     | -     |
|            | V053                                     | 48.72                 | 5.00                                  | 53.75                            | 50.63   | 50.07 | 50.05 | 50.06 | 50.05 | -     | -     |
|            | amount of bitumen in bitumen coating [%] |                       |                                       |                                  | 63.4  | 74.1  | 74.9  | 75.0  | 74.9  | -     | -     |

The results show that the calcination time for sample V05 was 2.33 h  $((2 + 2.5 + 2.5)/3)$ , for the test piece V10 was 2.5 h  $((2.5 + 2.5 + 2.5)/3)$  and for the test piece V15 was 3.67 h  $((3.5 + 3.5 + 4.0)/3)$ . The calcination time of 20 g sample, which was determined by regression analysis, was 5.84 hours, see Fig. 5. The amount of bitumen mass found in V05 and V10 exceeded 72 %, in the case of V10 0.7 % and in the case of V05 2.9 %.

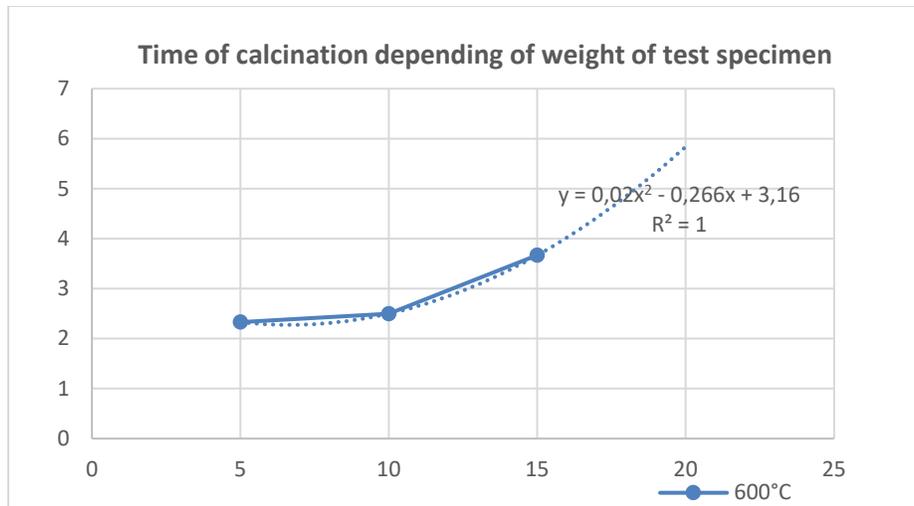


Figure 5. The time of the calcination depending on the weight of the test specimen

### 3.2. Test calcination of bitumen sheet - results of optimization of weight, size and arrangement of bitumen sheet test specimens in the incinerating dish

Table 2. Influence of the arrangement of the test specimens of the bitumen sheet in the incinerating dish on the time of the calcination

| Test piece | Type of dish    | The number of layers of bitumen sheet | Weight of test specimens | Total size of the test part of test specimen | Size of individual parts of the test specimen | Time of calcination |
|------------|-----------------|---------------------------------------|--------------------------|--|---|---------------------|
|            |                 | [piece]                               | [g]                      | [mm]   | [mm]  | [h]                 |
| V01        | deep dish DN 71 | 2                                     | 14.06                    | 50 x 50                                      | 25 x 50                                       | 3.5                 |
| V02        | deep dish DN 71 | 4                                     | 26.73                    | 50 x 100                                     | 25 x 50                                       | 4                   |
| V03        | low dish DN 81  | 1                                     | 13.04                    | 50 x 50                                      | 50 x 50                                       | 1.75                |
| V04        | low dish DN 81  | 3                                     | 27.21                    | 50 x 100                                     | 2 x 25 x 50 + 50 x 50                         | 3.5                 |

The results show that the calcination is faster using a low dish for the sample of bitumen sheets of similar weight. However, due to the low height of the dish, not all the matter remained inside during the calcination. The relocation of a low incinerating dish with the help of laboratory crucible tongs is bad. It is necessary to hold the dish by the edge, where there is also a part of the calcinated mass. Thus, a high dish DN71 mm was chosen as the optimal one.

### 3.3. Results of calcination of bitumen sheet

Table 3. The overview of results of the calcination of the bitumen sheet. Source: Own.

| Sample | Test specimen                       | Time of calcination [h] |      |      |      |      |      |      |      |      |      |
|--------|-------------------------------------|-------------------------|------|------|------|------|------|------|------|------|------|
|        |                                     | 1                       | 1.5  | 2    | 2.5  | 3    | 3.5  | 4    | 4.5  | 5    | 5.5  |
| BS     | BS1                                 | 2501                    | 2554 | 2641 | 2737 | 2822 | 2900 | 2954 | 2989 | 2995 | 2997 |
|        | BS2                                 | 2467                    | 2503 | 2584 | 2679 | 2765 | 2850 | 2915 | 2953 | 2956 | 2956 |
|        | BS3                                 | 2489                    | 2537 | 2611 | 2691 | 2769 | 2842 | 2908 | 2949 | 2973 | 2977 |
|        | arithmetic mean [g/m <sup>2</sup> ] | 2485                    | 2531 | 2612 | 2702 | 2785 | 2864 | 2925 | 2964 | 2975 | 2977 |

The amount of combustible residues in the bitumen sheet was 2 977 g/m<sup>2</sup>. Due to the fact that the carrier was made of polyester and the bottom surface treatment consisted of an easily combustible PE + PP foil, the weight of the amount of the two materials were subtracted. The amount of the bitumen mass in the bitumen sheet was determined to be equal to the following value 2757 g/m<sup>2</sup> (= 2977 - 200 - 20).

**3.4. Results of extraction of bitumen sheet and bitumen coating material**

Table 4. Mass of the bitumen sheet and percentage of bitumen mass in the bitumen coating mass

| Test specimen   | Bitumen sheet [g/m <sup>2</sup> ] | Bitumen mass [%] |
|-----------------|-----------------------------------|------------------|
| 1               | 2763                              | 73.3             |
| 2               | 2705                              | 72.3             |
| 3               | 2754                              | 72.7             |
| arithmetic mean | 2741                              | 72.7             |

**4. Discussion of results**

**4.1. Calcination of bitumen coating material**

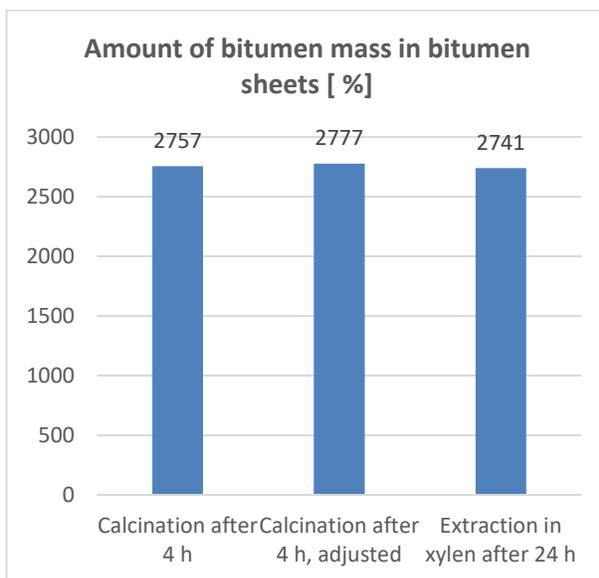
The calcination time for the expected limit of the mass of bitumen coating material was between values 3.67 and 5.84 hours. The calcination of the bitumen sheet took place in 4.8 h (= (4.5 + 5 + 5)/3) until the weight has stabilized. The result was obtained in the expected interval.

**4.2. Test calcination of bitumen sheet - results of optimization of weight, size and arrangement of bitumen sheet test specimens in the incinerating dish**

The first hypothesis asserting that the weight, size and arrangement of the test specimen in the incinerating dish have an effect on the time of duration of the calcination has been confirmed. When comparing the same area of the test specimen, the time of the calcination was the shortest using a low dishes. The sample was better accessible for thermal processes. The effect of the number of layers on the time of calcination was more pronounced for a smaller test specimen with an area of 50 x 50 mm.

**4.3. Comparison of calcination and extraction of bitumen coating**

The third hypothesis asserting that the differences between the results of individual methods (extraction and calcination) for the sample of bitumen coating material will be up to 2 % has been confirmed. Both methods report a very comparable result, see Figure 6.



The small difference of 0.7 % of the mass in bitumen in the results of both methods is due to the fact that the test specimens had the same weight. The difference between the recipe and the results is probably due to the preparation of a small sample. In laboratory conditions, it is problematic to prepare a large sample. The difference in the results can be caused by the sedimentation of the fillers or by the adhesion of some of the fillers and the polymer to the walls of the vessel in which the mass is prepared.

Figure 6. The comparison of the mass in the bitumen coating mass

#### 4.4. Suitability of the test procedure according to the prepared standard prEN 544:2020

The second hypothesis is declaring: It will not be possible to use the test procedure according to prEN 544:2020 [8] for bitumen sheets. The time of calcination of 2 hours according to [8] will not be sufficient for bitumen sheets. This hypothesis has also been confirmed. The test procedure in [8] cannot be used due to the fixed calcination time of the test specimen. The test specimen measuring 50 x 100 mm contains less bitumen coating materials than the bitumen sheet according to [3]. This fact is based on the requirements for the minimum amount of bitumen. According to ČSN EN 544 [16] bitumen sheet contains at least 1 300 g/m<sup>2</sup> for single-layer shingles and 1 500 g/m<sup>2</sup> for multi-layer. The bitumen sheet contains at least amount of bitumen 2 500 for base sheets and final layer 4.0 and 4.2 mm thick. Double time of the calcination corresponds to double amount of bitumen mass for bitumen sheets.

#### 4.5. Comparison of results of the calcination of the bitumen sheet with the results of extraction of the bitumen sheet

The third hypothesis assuming that the differences between the results of individual methods (extraction and calcination) for the sample of bitumen sheet will be up to 2 % has been confirmed. Thanks to the extraction, the actual basis weight of the carrier was determined. The mass of the carrier was 180 g/m<sup>2</sup>. In this case, the actual mass of bitumen of the carrier liner was 20 g lower than expected. The actual mass of the bitumen was thus 2977 - 180 - 20 = 2777 g/m<sup>2</sup>, see Fig. 7. The amount of bitumen mass determined by the calcination was higher than by the extraction method. The difference between the results obtained using the calcination and the extraction was 36 g, which represents 1.3 % (2777 - 2741 = 36 g/m<sup>2</sup>).

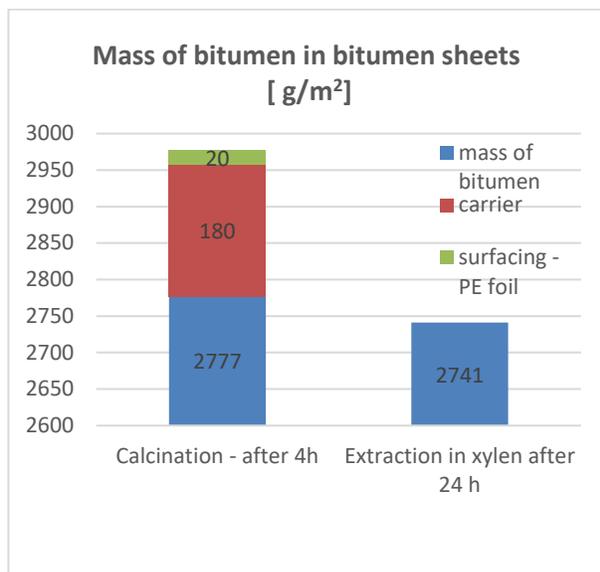


Figure 7. Comparison of the amount of bitumen mass in the bitumen sheet

Assuming that the extraction is a more accurate method - other non-combustible residues such as the carrier and polymer film are retained, so if we subtract the maximum expected basis weight of the carrier (250 g/m<sup>2</sup>) and polymer film (20 g), the total amount of bitumen mass must be at most equal to the extraction result. It will be about at least the amount of bitumen mass.

The difference in results is greater than in the case of the testing bitumen coating. The difference is due to the different weights of the individual test specimens. The difference is even though the test specimens are close to each other. Homogeneity of the bitumen coating material, uniform thickness as well as uniform spreading are never fully ensured in the production of bitumen sheets.

#### 5. Conclusion

All three hypotheses have been confirmed. During the tests of the bitumen sheet, it has found that the time of the calcination for the bitumen sheet 4.2 mm thick with elastomeric modification of bitumen mass is about 4 hours. We recommend the end of the calcination if the weight loss of the test specimen was less than 0.03 g. The time of the calcination of the bitumen sheet and the bitumen coating material, with a comparable amount of bitumen material, differed by 0.5 h. The difference in the results of both methods depends not only on the estimation of the mass of combustible residues (carrier, separation and coating foils), but also on the different basis weights of the test specimens. The bitumen sheet is not homogeneous in width or length from the principle of the production. The difference between two methods

is given by the different thickness and the mass of the test specimens. The calcination method is as the time consuming as the extraction method for bitumen sheets with bitumen coating material of elastomeric character. The calcination of the bitumen sheet meeting the requirements [3] takes 4 - 6 hours depending on the amount of bitumen mass, while the extraction takes about 12 - 14 hours [1]. We assume that the calcination method may become a new valid test method in the future.

The subject of further research would be turned to the elaboration of the calcination methodology using several samples and test specimens and shortening the calcination time. The paper has been developed with the support of the project SVV082021.

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