

Determination of mass of bitumen in bitumen membranes

J. Plachý, T. Navara

Institute of Technology and Business in České Budějovice, Faculty of Technology, Department of Civil Engineering, Czech Republic

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Abstract

Bitumen membranes have a primarily waterproofing function in the construction industry. The waterproofing function in bitumen membranes is provided by the mass of bitumen. The amount of bitumen mass can be determined by various methods. This paper deals with the determination of the bitumen mass in bitumen membranes by means of extraction and calcination. In the current legislation, the quantity of bitumen mass can only be determined by extraction. The calcination method is specified in the forthcoming EN 544, which is used for bitumen shingles. Previous research by the author has shown that the methodology from this standard cannot be used. The aim of this paper is to provide a practical verification of the extraction and calcination methods from previous author's research on a set of selected bitumen membranes. A total of 6 bitumen membranes were used for comparison. The bitumen membranes were of the same type from different manufacturers. The bitumen membranes were extracted in xylene. Calcination was carried out at 600°C. Two claims were made that confirmed the results of previous research on the bitumen coating and bitumen membranes. The first claim was that the calcination method is as time consuming or faster. This claim was confirmed. The second claim was that the results between extraction and calcination would differ by a maximum of 1.3%. The second claim was only partially met because the two asphalt strips contained insoluble but burnable parts. The results therefore differed by more than 1,3 %.

Keywords: *Calcination; Extraction; Mass of bitumen; Bitumen membranes; Bitumen sheets; Bitumen coating; Bitumen sheets for underlay; Bitumen area weight; Incinerating dish.*

1. Introduction to the Issue- why determine the area weight of bitumen (mass of bitumen) on bitumen membranes?

Bituminous membranes in the construction industry have primarily a waterproofing function. Nowadays, in an attempt to reduce the price of bitumen membranes, the amount of fillers is increased, the amount of bitumen is reduced and the lifetime of waterproofing is shortened [1-4]. This paper deals with the problem of determining the mass of bitumen in bituminous membranes that provides the waterproofing function. The work deals with the practical verification of the results and is directly related to papers [1-2]. Bitumen membranes are manufactured according to European product standards, where the requirement for the amount of bitumen is not specified. In the case of the Czech Republic, the procedure for the determination of bitumen in bituminous membranes is given in ČSN 730605-1, Annex D [5]. The amount of bitumen is determined here by Soxhlet extraction. This issue is dealt with in more detail in [1]. Another way to determine the mass of bitumen in bituminous membranes is by calcination. According to the information available to us (as of 12/2023), the calcination method is not yet described in any of the test codes that deal with the determination of the mass of bitumen in bituminous membranes. The calcination method is only addressed in the forthcoming asphalt shingle standard EN 544 [6]. This issue is dealt with in more detail in [2].

This work aims to perform a practical verification of the extraction and calcination from the authors' previous research [1-2] on a set of bitumen membranes. For the verification, 6 samples of self-adhesive bitumen membranes with a thickness of 3 mm and a SBS cover and a glass

fibre support were used. The results of the bitumen area weight in bitumen membranes obtained by extraction and calcination method will be compared.

Two statements have been established based on confirmed hypotheses from publications [1-2]. The first claim. The calcination method is faster in terms of time than the extraction method. This claim was confirmed. Second claim. The differences between the results of the different methods (extraction and calcination) for asphalt strip samples will be up to 1.3 %. The 1,3 % limit was chosen for the reason given below. In the sense of products, the term bitumen membranes is used. In the sense of tests, the term bitumen sheets.

The quantity of bitumen mass for the bitumen sheets for underlay thickness of 3.0 mm used for roof waterproofing according to CSN 730605-1:2014 [5] is 1500 g/m². The usual area weight of the glass fibre liner for bitumen membranes is 160-200 g/m² according to our experience and according to [7-9]. Thus 180 g/m² +/- 20 g/m². The 20 g represents 1.3 %.

2. Material and methodology

2.1. Methodology – test procedure

The work can be divided into four stages. Stage 1. Preparation of test specimens and test piece. Stage 2. Calcination of the bitumen membranes. Stage 3. Extraction of the bitumen membranes. Stage 4. Evaluation

2.1.1. Preparation of test specimens and test piece

The preparation of test specimens for extraction and calcination is based on [1-2]. For extraction and calcination, three test specimens of the same size of 100 mm x 100 mm were used. The same size is for the sake of comparing the results of the bitumen mass between the two methods. The test specimens were taken along the width of the bitumen sheets perpendicular to the production direction. The test specimens for calcination were selected from the same sample (test piece) as for extraction. See Figure 1 for a sample of bitumen sheet with the indication of the location of the test specimen and test pieces. See Figure 2 for the position of the test pieces. Part of the preparation of the test pieces was the thickness measurement according to EN 1848-1 [10].

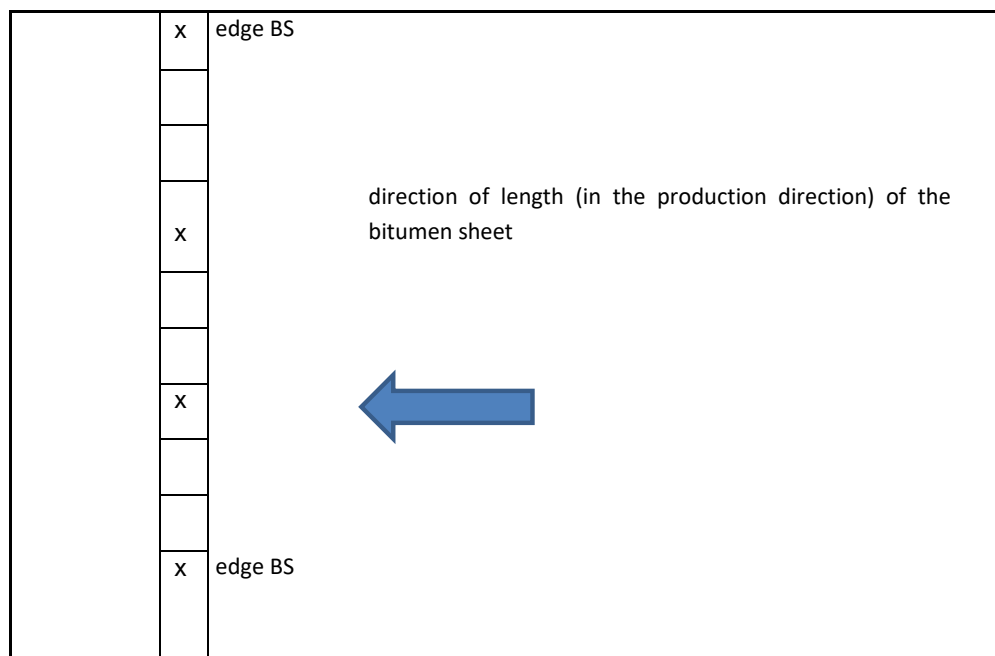


Figure 1. The sample of bitumen sheet with the indication of the location of test pieces. Source: [2].

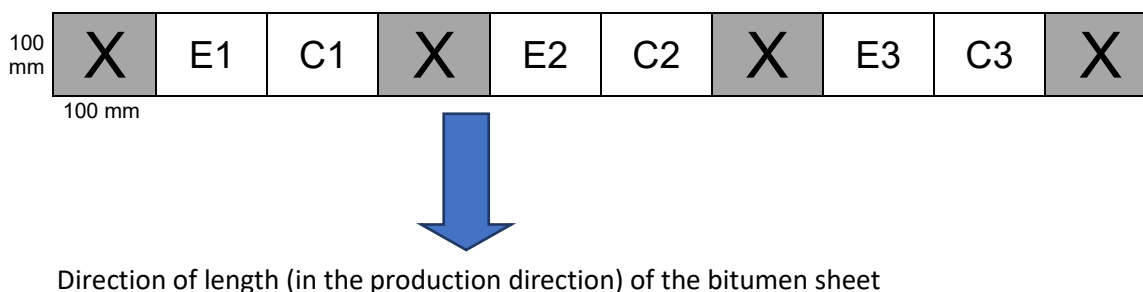


Figure 2. The location of test specimens in the test piece of bitumen sheet. E1, E2, E3 – test specimens for extraction, C1, C2, C3 – test specimens for the calcination. Source: [2].

2.1.2. Calcination of the bitumen sheet

The test procedure for calcination was based on the modified prepared ČSN EN P 544 [6] and on the results according to [2]. According to this methodology, the test is carried out on three test specimens. The temperature in the furnace during calcination is 600 °C. Weighing accuracy 0,01 g. Incinerating dish-deepform vessels of 35 mm height and 70 mm diameter were used as calcination vessels. See Figure 3. All three test specimens were tested simultaneously. The number of Incinerating dish was six. Therefore, it follows that one 100 x 100 mm test specimens will be divided into two Incinerating dish. Thus, there is half of a 50 x 100 mm test specimens per bowl.

The test was terminated only when the weight loss was less than 0.03 g [1]. The weight loss was monitored at 0.5 h intervals with the first measurement after 3 h. The resulting time for the end of firing will be calculated as the arithmetic mean of the times from the measurements of the three test specimens.



Figure 3. Incinerating dish, deep form. Source: [2].

To determine the amount of bitumen mass in the bitumen sheets, it is necessary to subtract other burnable residues that are not part of the bitumen mass from the obtained area mass. These are mainly polymer films and carrier. The bitumen sheet tested had a removable polymeric film. The usual area weight of the removable film is 30-40 g/m² [7-9]. The actual areal weight of the removable film was verified by weighing it on a laboratory balance. Since the bitumen sheets have a glass fabric reinforcement, there is no subtraction of the area weight of the reinforcement liner from the observed area weight. Other burnable residues may be separation films on the top or bottom surface of the bitumen sheets. The area weight of the separation and protective polymeric film is typically 20 g/m² [7,11].

2.1.3. Extraction of bitumen sheet

The test procedure for extraction is based on [1-2]. The test is performed on three test specimens. The test specimens were inserted into the extractor folded in filter paper. The test was terminated when there was colorless solvent in the filter paper extension.

2.2. Materials – bitumen membrane

The bitumen membrane with a thickness of 3 mm, which is used as a base layer for roof waterproofing, was selected for the tests to determine the bitumen mass. It was a self-adhesive bitumen membrane with a material modified with elastomeric polymers (SBS). Elastomeric polymers were chosen because this type of modification is the most frequently used in the conditions of the Czech Republic [1-2,8,12]. The carrier of the bitumen membrane was made

of glass fabric with an area weight of 180 +/- 20 g/m². See Table 1 for the specifications of the bitumen membrane.

Table 1. Overview of used bitumen membranes samples. Source: Own.

signification / monitored properties	VZ1	VZ2	VZ3	VZ4	VZ5	VZ6
top surface treatment			fine-grained mineral slate			
bottom surface treatment			easily combustible foil			
bitumen mass modification			PE + PP			
carrier			elastomeric			
			glass fiber			
mass of the carrier g/m ²	180	180	180	180	180	180

2.3. Extraction materials - solvents

Based on previous research [1], xylene was used as the solvent. In terms of quality (purity of the chemical), the solvent used for the analysis was (p.a.)

2.4. Test equipment - tools and instruments

The following apparatus, aids and materials were used for the tests. For the extraction, apparatus according to [1-2,5] were used. Namely, an extraction apparatus according to Soxhlet with a 500 ml attachment, a heating nest with a volume of 2000 mL, a balance with an accuracy of 0.01g, a drying oven with forced air circulation. Electric furnace.

2.5. Evaluation methodology

The results of the area weight of bitumen after extraction will be compared with the results of the area weight of bitumen after calcination. The extraction results were considered to be more accurate due to the fact that not all of the burnable parts are soluble. It is considered that an extraction time of 24 h according to [1] is sufficient. The basic assumption for the comparison is that the difference in thickness of the compared test specimens will be within 0.05 mm.

If the differences between the results of the different methods (extraction and calcination) are within 1,3 %, the result can be considered excellent. If the result is higher, it will be necessary to adjust the bitumen mass loss limit of 0.03 g, to review the area weight of the bitumen membranes carrier, to recalculate the results to the same area weight of the bitumen sheets test specimen.

3. Results

3.1. Results of calcination and thickness measurements of bitumen membrane samples

The calcination of all bitumen membrane samples was between 5 - 5.5 h. See Table 2 for a summary of the results of the asphalt area weight.

Table 2. Summary of the results of the asphalt area weight obtained by the calcination method. Source: Own.

Sample /Test body	VZ1		VZ2		VZ3		VZ4		VZ5		VZ6	
	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]
C1	2.96	2089	2.93	2081	2.75	2029	2.85	1987	3.08	1900	3.01	1953
C2	3.17	2217	2.70	1880	2.75	2079	2.79	1984	3.14	2074	2.93	1862
C3	2.96	2085	2.79	2029	2.88	2077	2.87	1926	2.99	2000	2.82	1739
AM [g/m ²]		2130		1997		2062		1966		1991		1908
PE films [g/m ²]		42		30		30		30		35		30
AM [g/m ²]	3.03	2088	2.81	1967	2.79	2032	2.84	1936	3.07	1956	2.97	1878

Legend: th. – thickness, MOF – mass of bitumen, AM – arithmetic mean.

3.2. Extraction results and thickness measurements of bitumen membrane samples

Extraction of all bitumen membrane samples was between 9 - 16 h. See Table 3 for a summary of the results of the asphalt area weight.

Table 3. Summary of the results of the asphalt area weight obtained by the extraction method. Source: Own.

Sample/ Test body	VZ1		VZ2		VZ3		VZ4		Vz5		VZ6	
	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]	th. [mm]	MOF [g/m ²]
E1	2.84	2028	2.89	1955	2.83	1818	2.84	1789	3.03	1814	3.02	1913
E2	3.23	2230	2.76	1928	2.65	1754	2.87	1725	3.12	1993	2.94	1801
E3	2.92	2049	2.79	1957	2.78	1843	2.94	1758	3.14	2025	2.77	1671
AM [g/m ²]	3.00	2102	2.81	1947	2.75	1805	2.88	1757	3.10	1944	2.98	1857

Legend: th. – thickness, MOF – mass of bitumen, AM – arithmetic mean.

4. Discussion of results - Comparison of bitumen membrane calcination results with bitumen membrane extraction results

The **first statement** that the calcination method is faster than the extraction method was confirmed for all bitumen membrane samples. Calcination of bitumen membrane test samples was between 5 - 5.5 h. Extraction of bitumen membrane test samples was between 9-16 h. The research findings [1-2] were confirmed.

The **second claim** that the differences between the results of the different methods (extraction and calcination) for the bitumen membrane sample would be up to 1.3% was only partially confirmed. See Figure 4. Two of the six samples showed burnable but insoluble parts. Through extraction, it was determined that these were probably rubber. Therefore, objective results were only available for four asphalt strips. The amount of asphalt mass detected by calcination was higher in three of the four bitumen membrane samples. This can be explained by the fact that some insoluble parts are burnable.

The results of the research pointed to a further direction of research. These are mainly questions on how to work (interpret) with burnable residues that are not soluble. Furthermore, how to approach test specimen that differ significantly in thickness or areal weight. The bitumen membrane is not homogeneous in width or length by the principle of manufacture. Thus, the difference between the two methods may be due to the different thicknesses and specific weights of the test specimen. Thus, the test specimen are different.

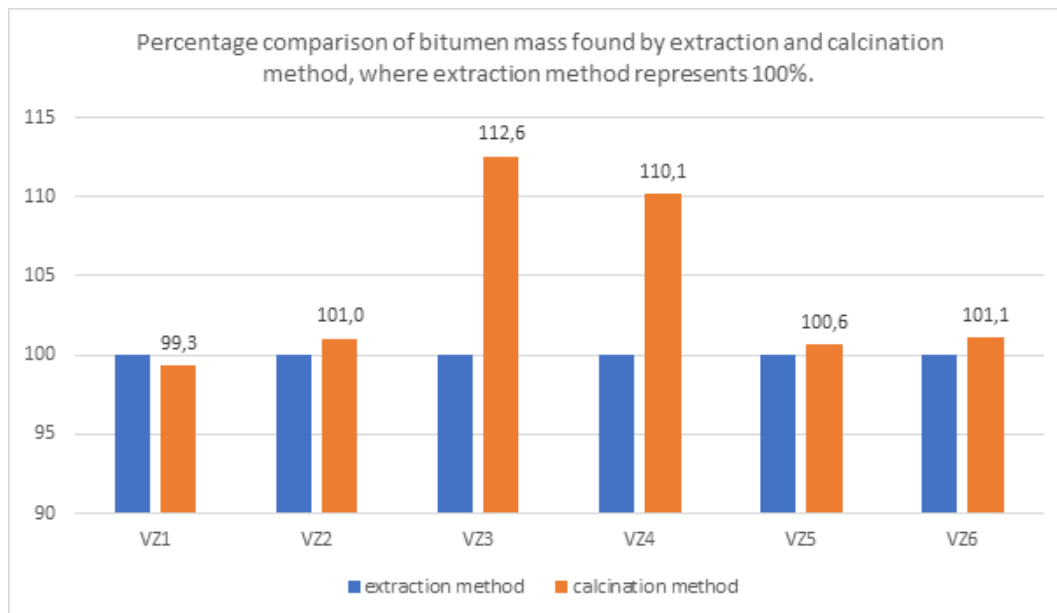


Figure 4. Comparison of bitumen mass in bitumen membranes. Source: Own.

5. Conclusion

To verify the results of the previous research, bitumen membranes with non-burnable bearing inserts were used. The use of non-combustible carrier reduced the possible error in comparing the results of the bitum area weight obtained by calcination and extraction methods.

The claim that the calcination method is less time consuming than the extraction method for bitumen membranes with elastomeric bitum overlays was confirmed. The second claim that the differences between the results of the different methods (extraction and calcination) for the bitumen web sample would be up to 1.3% was only partially confirmed. Two of the six bitumen membranes contained larger amounts of insoluble but burnable residues.

In the bitumen membranes tests, the burning time (time) for a 3.0 mm thick bitumen membranes with elastomeric bitum modification was found to be approximately 5.0 - 5.5 h. The time required for extraction of the bitumen membranes was 9-16h. Further, the calcination was confirmed to be complete if the weight loss of the test specimen is less than 0.03g.

We still expect that the calcination method will become a new valid test method for determining the bitumen mass in bitumen membranes. However, it can be said that new definitions will have to be used in the future. Instead of the bitum area weight, define terms such as the proportion of combustible and the proportion of extractable parts in bitumen membranes.

The subject of further research would be to refine the calcination methodology on more samples with flammable reinforcement carriers.

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To whom correspondence should be addressed: Dr. J. Plachý, Institute of Technology and Business in České Budějovice, Faculty of Technology, Department of Civil Engineering, Czech Republic, E-mail: plachy@mail.vstecb.cz