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Determination of Mass of Bitumen in Bitumen Sheets by Extraction

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Abstract

The work deals with the problem of determination of mass of bitumen (bitumen polymer) in bitumen sheets. The component influencing waterproofing ability is bitumen. For this reason, in some countries there are defined requirements for the mass of bitumen in bitumen sheets. This contribution shows the possibilities of how to determine the mass of bitumen in bitumen sheets. The methodology for determining mass of bitumen is based on a comparison of test procedures specified in the standard ČSN 730605-1 and DIN 52123, where the mass of bitumen is determined by extraction and indicates the insufficiency in current legislation where the extraction method is not sufficiently described. The work specifies the extraction method by precisely defining aids and procedures and focuses on the possibility to replace trichloroethylene with another solvent in order to limit the use of this solvent. The work compares the measured results of mass of bitumen for two samples of bitumen sheets (modification by plastomers and modification by elastomers) in for three selected solvents (trichloroethylene, xylene and toluene). Mass of bitumen was determined always within 24 hours. The results are comparable with the following specification. Sheets with Styrene-butadiene-styrene (SBS) polymer show a smaller standard deviation and xylene more accurate results in the relation to trichloroethylene. Economic costs for extraction in the case of xylene represent 331 % of the costs and in the case of toluene it has reached 158 % costs of the compared to using trichloroethylene. Xylene and toluene are comparable in terms of the medical harmlessness. The work is in the first phase of the research, which in the next stage will deal with another way to determine bitumen in bitumen sheets by using of the calcination.

***Keywords**: Solvent; Extraction; Bitumen sheets; Mass of bitumen.*

1. Introduction - why to use extractions for bitumen sheets, is that the only way?

This contribution deals with determining mass of bitumen in bitumen sheets. Bitumen sheets primarily have a waterproofing function in the construction industry. The waterproofing function in bitumen sheets is provided by bitumen. Bitumen sheets are produced according to European product standards, which are divided according to of their using. Therefore, we can sort bitumen sheets used for roofs (ČSN EN 13707 [1]), for substructures (ČSN EN 13969 [2]), for bridge insulation (ČSN EN 14 695 [3]) inserted into wall structures (ČSN EN 14967 [4]), for vapor barriers (ČSN EN 13970 [5]) and for a base for folded coverings (ČSN EN 13859-1 [6]) or wall sheets (ČSN EN 13859-2 [7]). Requirement for the mass of bitumen in bitumen sheets in named standards is not stated. Some EU countries are not satisfied with this situation and so they require the determination of the mass of bitumen in their national standards. These are, for example, the Czech Republic and the Federal Republic of Germany.

In the case of the Czech Republic, it is the standard ČSN 730605-1 [8] determining the mass of bitumen in bitumen sheets, which are produced according to ČSN EN 13707 [1], ČSN EN 13969 [2] ČSN EN 13 970 [5]. Requirements for the mass of bitumen in the Federal Republic of Germany are given in several standards DIN SPEC 20000-201 [9], DIN SPEC 20000-202 [10] and DIN V 20000-203 [11]. It is important for determining mass of bitumen in bitumen sheets according to which methodology the tests are performed. The methodologies are distinct and they differ from each other. In the case of the Czech Republic the test procedure for the determination of mass of bitumen in bitumen sheets is included directly in ČSN 730605-1 [8], Annex D and in the case of the Federal Republic of Germany the procedure is directly described in DIN 52123: 2014 [12].

In both cases, the mass of bitumen is determined by extraction. Extraction is a separation process in which substances contained in one phase of the material are divided into other phases on the basis of different solubility in the solvent used. In both standards [8,12] Soxhlet extraction is used. It is a continuous solid-liquid extraction (SLE).

In practice, however, it is possible to determine the mass of bitumen in bitumen sheets by two methods, namely extraction or calcination. In addition, the newly prepared standard for bitumen shingles EN 544 [13] is considering the calcination method. The method of calcination for sheets is the subject of further research.

Both standards [8,12] list several solvents, but only three are common, of which trichloroethylene can be considered the solvent with the highest efficiency and especially in the Czech Republic was and is very often used [14-15]. However, according to Regulation (EC) No. 1272/2008 of the European Parliament and of the Council of 16 December 2008 on the classification, labeling and packaging of chemicals and mixtures, the so-called "CLP Regulation", and the solvent is classified as "Harmful". EC regulations classified it as "toxic" (T). According to the REACH Regulation (Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorization and Restriction of Chemicals), trichloroethylene is classified as a carcinogen, category 1B. Pursuant to Commission Regulation (EU) No 348/2013 of 17 April 2013 amending Annex XIV to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH), trichloroethylene in the industrial production was to be replaced by another substance from 21 April 2016, but the use was extended [15].

The aim of this work is to find a new solvent instead of used trichloroethylene. The selection has been made on the basis of a comparison of selected types of solvents in terms of determining the mass of bitumen, the time of extraction-separation (dissolution), economic costs and occupational safety. Three basic hypotheses were established. The extraction of selected bitumen sheets will take place to 24 hours. Supposed that the mass of bitumen in the newly selected types of solvents will differ from the amount determined by using trichloroethylene by a maximum of 5 % and the economic costs between the individual solvents will differ from the costs related to using trichloroethylene by a maximum of 100 %. According to the available information, no one dealt with comparing the effectiveness of individual solvents for bitumen sheets. The issue of bitumen extractions was mentioned only by authors dealing with the extraction of bitumen by solvents from mineral deposits of oil sand [16] and the composition of bitumen, for example [17-18].

2. Material and methodology

2.1. Extraction methodology - test procedure

The extraction test methodology was based on a comparison of test procedures stated in [8] and [12]. Some specifics have been added from the standard ČSN 503602 [19]. The overview of the solvents used is included in Table 1. The selection of test specimens (TS) was carried out in accordance with the standard DIN 52123. A total of three test specimens measuring 50 x 100 mm were taken from the edges and center of the bitumen sheets and the test specimen was situated in the distance of 100 mm from the edge of the bitumen sheet. TS were conditioned per 2 hours 23°C with deviations ±2°C. An extraction apparatus according to Soxhlet, of volume 500 mL and cellulose cartridges with a round bottom 32 x 100 mm were used for the extraction itself. In each cartridge there was one test body closed with cotton wool. Before transferring the solvent from the nozzle to the flask, the temperature in the nozzle and in the flask was measured with a pyrometer (non-contact infrared thermometer). The test was terminated when there was a colorless solvent in the cartridge attachment. After cooling, the TS cartridge was placed in a fume hood for 30 minutes where part of the solvent was evaporated (released). Subsequently, the cartridge with the test body was placed in an oven at 105°C. The cartridge was weighed at hourly intervals until a weight gain of less than 0.03 g was obtained. The value of 0.03 g was chosen because it represents 0.1 % of the assumed mass of the bitumen in the bitumen sheets. Values of mass of bitumen move usually in the limits interval of 2500 - 2900 g/m^2 [8].

Table 1. Comparison of individual solvents used in ČSN 730605-1 [8] and DIN 52123 [12]

Type of solvent/standard	ČSN 730605-1	DIN 52123
Toluene (methylbenzene)	X	X
Xylene (dimethyl benzene)	X	X
Trichloroethylene (trichloroethene)	X	X
Methylene chloride (dichloromethane)	X	-
Perchlorethylene (tetrachlorethene)	X	-
Chloroform (trichlormethane)	-	X

Legend: X means contains, - means does not contain. Source: own

2.2. Materials for extraction – bitumen sheets

Bitumen sheets (BS), which are used in the construction, are composite materials. BS are composed of an upper surface treatment, an upper bitumen covering material, load-bearing inserts, a lower bitumen covering material and a lower surface treatment. Two types of sheets BS have been selected for the testing of the mass of bitumen, which differ only in the type of upper and lower bitumen covering material and thickness.

For bitumen covering material is used oxidized bitumen or bitumen treated with modifiers. Polymers of elastomeric or plastomeric character are used as modifiers. For bitumen sheets labeled by SBS Styrene-butadiene-styrene polymer was used as the bitumen modifier. For bitumen sheets marked APP, polymers IPP, PP, and APAO were used as modifiers. The used primary bitumen was produced from URAL crude oil by vacuum distillation. The specification of BS is included in Table 2.

Table 2. The overview of used bitumen sheets

Monitored properties/ signification	Bitumen sheet SBS	Bitumen sheet APP
Thickness	5.2 mm	5.0 mm
Top surface treatment	coarse-grained mineral gritting	coarse-grained mineral gritting
Bottom surface treatment	easily combustible PE+PP foil	easily combustible PE+PP foil
Modification of bitumen mass	elastomeric	plastomeric
Carrier	PES	PES
Mass of the carrier	220 g/m^2	220 g/m^2

Source: own

2.3. Materials for extraction - solvents

Based on [8] and [12], three solvents have been selected for comparison. These are toluene, xylene and trichloroethylene. In terms of the quality (chemical purity), solvents have been used for the analysis (p.a.) [20]. The content of the basic substance is moving usually between values 99.0-99.8 %. In terms of relative permittivity, toluene and xylene are close situated together in an elastotropic series of solvents. Thus, a similar result can be expected in terms of the mass of bitumen and the time of cleaning, in the contrast to results obtained for trichloroethylene, which has a higher relative permeability. An overview of selected properties of used solvents is completed in Table 3.

2.4. Testing equipment - aids and devices

The following aids and materials were used for tests: Soxhlet extraction apparatus with 500 mL attachment, 2000 mL heating nest, round-bottom pulp extraction cartridges 32 x 100 mm, cotton wool, balance with an accuracy of 0.01 g, forced-air oven, extractor hood, pyrometer (infrared) thermometer) Reitek with emissivity E=0.93, protective equipment-goggles, gloves.

Table 3. The overview of used solvents and their selected properties

Business name	Units	Toluene	Xylene	Trichloroethylene
Chemical name		methylbenzene	Dimethyl benzene	trichloroethene
Viscosity	(mPa·s at 20°C)	0.59	not available	not available
Density	(g/cm^3 at 20°C)	0.866	0.867	1.46
Boiling point (at 101,325 kPa)	°C	110	135-143	87
Steam tension	hPa	31	8	213
Relative permittivity		2.3 - 2.38	2.2 - 2.5	3.4
LC_{50} by inhalation	mg/L	29	27	12500 ppm/4h (sewer rat)
LC_{50} orally	mg/kg	5580 (rat)	4300 (rat)	4290 (sewer rat)
PEL (8 hour exposure)	mg/m^3	200	200	250
NPK-P (15 minutes exposure)	mg/m^3	500	400	750

Source: own

2.5. Methodology of solvent evaluation

The evaluation has been performed by using multi-criteria analysis. The results should take into account both types of bitumen sheets. There have been stated four main criteria: mass of bitumen, the time of extraction, test costs and toxic safety. Based on our practical experience, the mass of bitumen gets the highest weight. The resulting arithmetic mean of the mass of bitumen in xylene and toluene will be compared to results while using trichloroethylene where mass of bitumen result of which will be considered here to be 100 %. Another criterion is the time of the extraction. The measurement result of mass of bitumen must be obtained quickly. Less weight is assigned to costs and only direct energy costs - electricity, water - will be reflected in the costs. The percentage comparison would be the same as for the time of extraction, because energy consumption is directly dependent on the time of extraction. Safety and toxicity will be of the lowest weight, as xylene and toluene are similar solvents. The LC_{50} parameter, the so-called Lethal Concentration 50, was chosen as the criteria for comparison, which expresses the mortal concentration for 50 % of the experimental animals. From the point of view of the work of the operator performing the tests, binding limits have been selected for the Czech Republic according to Government Decree No. 361/2007 Coll. Annex 2. This is the Permissible Exposure Limit (PEL) in mg/m^3. It must not be exceeded in an 8-hour shift. Another limit is the Maximum Permissible Concentration in the Working Air (NPK-P) [mg/m^3].

3. Results

Table 4. Determination of mass of bitumen

Type of solvent	Bitumen sheet		Cleaning time	Solvent temperature attachment	Solvent temperature flask	Mass of carrier	Total mass of bitumen
		Type of test specimen	Hour: minutes	(°C)	(°C)	(g/m^2)	(g/m^2)
Toluene	SBS	ZT1	14:10	63	93	301	3967
		ZT2	12:35	64	90	304	4220
		ZT3	13:55	69	93	299	4272
		AM	13:33	65	92	301	4153
	APP	ZT1	13:15	70	94	239	2897
		ZT2	10:30	62	92	264	2813
		ZT3	13:15	71	64	251	2813
		AM	12:20	68	83	251	2841

Type of solvent	Bitumen sheet		Cleaning time	Solvent temperature attachment	Solvent temperature flask	Mass of carrier	Total mass of bitumen
		Type of test specimen	Hour: minutes	(°C)	(°C)	(g/m²)	(g/m²)
Xylene	SBS	ZT1	13:50	71	118	287	4071
		ZT2	13:40	60	107	312	3953
		ZT3	13:25	71	116	287	4114
		AM	13:38	67	114	296	4046
	APP	ZT1	14:25	87	110	255	3099
		ZT2	19:00	65	107	247	3096
		ZT3	10:40	63	114	253	2794
		AM	14:41	72	110	252	2996
Trichloroetylene	SBS	ZT1	4:15	61	78	282	4030
		ZT2	3:50	73	76	275	4073
		ZT3	4:10	65	75	289	4123
		AM	4:05	66	76	282	4075
	APP	ZT1	8:00	58	74	233	3132
		ZT2	9:50	62	72	251	3020
		ZT3	7:50	60	73	238	3129
		AM	8:33	60	73	240	3094

Legend: AM - arithmetic mean. Source: own

4. Discussion

4.1. Determination of mass of bitumen by extraction - comparison of solvents

The highest value of mass of bitumen for SBS was obtained by extraction of toluene followed by trichloroethylene and finally xylene.

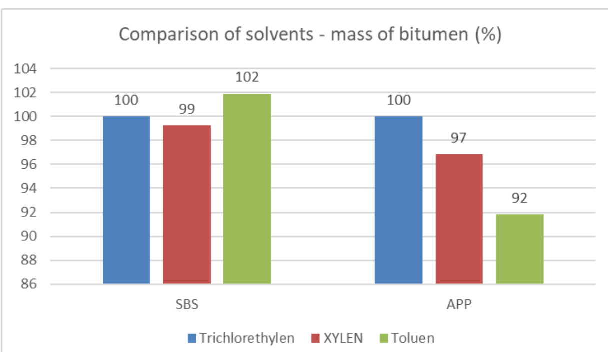

Figure 1. Comparison of solvents-mass of bitumen
Source: own

Measured results for individual solvents are comparable. The very low standard deviation in the value of 45 g/m^2 has been obtained for the SBS, for APP has reached the value of 107 g/m^2. The different result can be attributed to the different composition of the polymers in the bitumen mixture. Aromatic solvents toluene and xylene are more suitable for modifying SBS that contains aromatic styrene. If we consider mass of bitumen in bitumen sheets determined for SBS and APP while using trichloroethylene as 100 %, then with the exception of xylene for BSP, the deviations are up to 3 %, see Figure 1. In general, it can be stated that the use of xylene is more suitable than the use of toluene. Both solvents are more suitable for SBS. Therefore, if we should use only one solvent, xylene appears to be more suitable based on the results.

4.2. Test time

All tests were completed within 24 hours. For further research and comparison, it is therefore possible to set 24 hours as the limit time. The extraction time for SBS was up to three times longer for xylene and toluene than while using trichloroethylene. In the case of APP, the time of extraction was up to twice longer compared to the trichloroethylene time. The dissolution time corresponds to a comparable value of relative permittivity for toluene and xylene, especially for bitumen sheets with SBS. There is a more significant difference in APP. The different result can again be attributed to the different composition of the polymers in the bitumen mixture. The results show that trichloroethylene is clearly more effective for SBS than APP. If we consider the total extraction time for SBS and APP while using trichloroethylene to be 100 %, then the average time of both solvents for SBS is 331 % of the average trichloroethylene time. In the case of APP it is 158 % of the time while using trichloroethylene. If we evaluate the total time in terms of the type of solvent, then in the case of xylene it is 252 % of the time of trichloroethylene. In the case of toluene, this is 237 % of the trichloroethylene time. Detailed values are completed in Figure 2.

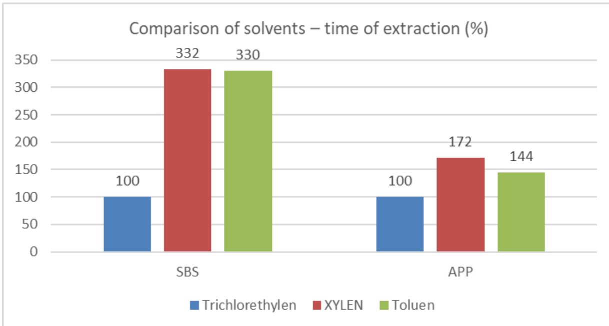

Figure 2. Comparison of solvents – time of extraction
Source: own

The drying (evaporation) time of all test specimens was up to 0.5 h, although trichlorethylene has the lowest boiling point, but at the same time it has the highest vapor pressure. From this point of view, the time when the test specimens are outside the dryer in the fume hood is important and the solvent is evaporating freely from the test specimens. The observed section of 0.5 hour would be too short, but from the overall point of view of extraction it is negligible.

4.3. Economic costs of the test

The economic costs of the tests are expressed by the price for the energy consumed in the average time interval of the test. The costs included electricity for the operation of the hood, the heating nest, dryer and water consumption for cooling, see Table 5. Energy prices are given currently for the Czech Republic: water - 90 CZK/m^3 [21], electricity - 5 CZK/kWh [22]. The cost of the test results depends on the time of the extraction. The price in the case of SBS is more than threefold in relation to use trichloroethylene and in the case of APP; it gives 1.5 times the price threefold in relation to use trichloroethylene.

Table 5. Economic costs of extraction

Electrical energy	Power consumption	Number	Total power consumption	Price	Price per hour of service
	kW			kW/h	CZK/h
Hood	0.11	1	0.11	4.8	0.528
Heating nest	0.48	3	1.44	4.8	6.912
Dryer	1.3	1	1.3	4.8	6.24
Water	Consumption	Number	Total power consumption	Price	Price per hour of service
	L/20s		m^3/h	CZK/m^3	
Flow through one cooler	1	3	0.54	100	54
Total consumption					67.68

Source: own

4.4. Occupational safety, information on toxic effects and exposure control

The criteria for comparison have been determined the information on toxic effects given in the safety data sheets [23-25], see values in Table 3. In the case of the LC_{50} parameter, a higher value is given for toluene than for xylene. Toluene is therefore less harmful. In the case of the PEL parameter, the values for toluene and xylene are the same. In the case of NPK-P, a higher value is given for toluene than for xylene. These values indicate that xylene is more toxic and harmful than toluene.

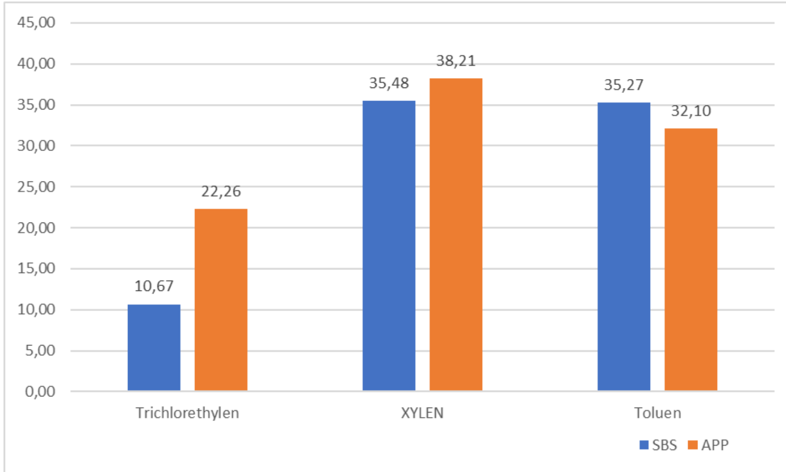

Figure 3. Comparison of solvents - energy costs for extraction of one sample (in Euro). *Source: own*

4.5. Multi-criterial evaluation of solvents

The evaluation has been performed according to a simple multi-criteria analysis, where the aspects are assessed: an accuracy of the value of the mass of bitumen, the time of extraction, economy and toxicity. Similar values have been measured for both solvents. The results show that xylene is more suitable, see Table 6.

Table 6. Multi-criterial evaluation of solvents

Business name	Weight	Toluene		Xylene	
Chemical name		methylbenzene		dimethylbenzene	
Type of bitumen sheet (BS)		SBS	APP	SBS	APP
Mass of bitumen	4	1	1	2	2
Time of extraction	3	1.5	2	1.5	1
Cost	2	1.5	2	1.5	1
Safety, toxicity	1	2	2	1	1
Total by solvent type BS		13.5	16	16.5	14
Total by solvent		29.5		30.5	

Source: own

5. Conclusion

Xylene can be used as an optimal replacement for trichlorethylene. However, the extraction time (purification) and thus the economic costs will increase. Only one hypothesis from three established hypotheses has been confirmed, two hypotheses have been partially confirmed.

For all three selected solvents (trichloroethylene, xylene, toluene) the extraction time was up to 24 hours. The hypothesis that the extraction will take place 24 h has been confirmed. The second hypothesis that the mass of bitumen in the newly selected types of solvents will differ from the amount determined by trichloroethylene by a maximum of 5 % has been only partially confirmed. In the case of toluene and APP sheets, the result determining the mass of bitumen differed by 8 %. In the case of xylene, the results determining the mass of bitumen differed by 2 % and 3 %. In the case of economic costs, the hypothesis that costs would increase by a maximum of 100 % has been again partially confirmed. For xylene, the cost accounted for 331 % of the cost of trichloroethylene. Concluded of results testing of toluene, where the cost was 158 % trichloroethylene, the hypothesis has been confirmed.

In the next phase of the research, we will deal with the determination of mass of bitumen by annealing (calcination).

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