Research and Measurement Status and Data Processing Technology of Highway Asphalt Intelligent Testing Equipment

Na Miao [a], Zhidan Lin [b], Ming Sun [c], Jing Zhu [d], Yixu Wang [e]

Research Institute of Highway Ministry of Transport Beijng, China

[a] n.miao@rioh.cn, [b] Linzd11@163.com, [c] m.sun@rioh.cn, [d] 530170517@qq.com, [e] wang.yx@rioh.cn

Abstract. This paper introduces the current status of the asphalt viscosity testing equipment in the field of the road engineering, domestic and foreign study about the high viscosity standard that is used for the calibration of such equipment, as well as the developing technologies of the viscosity standard; it also predicts and gives the outlook about the development trend of the high viscosity standard in the future.

Keywords: Road engineering; asphalt viscosity; testing equipment; high viscosity standard.

1. Introduction

The "Outline for Building China's Strength in Transportation" issued by the State Council of the People's Republic of China pointed out that "strengthening key technology research and development at the forefront, targeting the world's technological frontiers such as the next-generation materials, and enhancing the prospective, disruptive technology research triggering the transformation in the transportation industry". With the development of urban traffic in China, more than 90% of the urban road is the asphalt pavement by virtue of its characteristics such as providing comfortable driving. Asphalt is a macro molecular solution with the high-molecular- weight (HMW) asphaltene as the solute and low-molecular-weight (LMW) malthene (resin and oil component) as the solvent. Generally, the pavement properties of asphalt include viscosity, adhesivity, thermosensitivity, high-temperature stability, low-temperature crack resistance and aging resistance. Among them, the adhesivity of asphalt refers to both the physical adsorption capacity and chemical adsorption capacity that are generated by the interaction between the asphalt and aggregate. The adhesivity of asphalt and aggregate directly affects the quality and durability of the asphalt pavement, and, thus, adhesivity is one of the important indexes to evaluate the technical performance of asphalt[1]. At present, a lot of sets of asphalt viscosity testing equipment are used in the field of road engineering. The existing problem is that there is no production of the corresponding is cosity standard used for check and verification in China now. Besides the transportation industry, there are many industries, such as the petrochemical and electric power industries, with the testing need of higher viscosity points. The related enterprises can only buy these viscosity standards from abroad, and thus have to undertake a heavy economic burden, which results in difficulties in the technical performance determination of testing equipment.

Abstract: This paper introduces the current status of the asphalt viscosity testing equipment in the field of the road engineering, domestic and foreign study about the high viscosity standard for the calibration of such equipment, as well as the developing technologies of the viscosity standard; it also predicts and gives the outlook about the development trend of the high viscosity standard in the future, providing a certain reference for developing the high viscosity standard in the future.

2. Testing equipment of highway asphalt viscosity

At present, JTG E20-2011 Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering (hereinafter referred to as the Test Specification) is a specification formulated by the industry for standardizing and unifying the test methods of asphalt and asphalt mixtures and ensuring the quality of asphalt and asphalt mixtures in highway engineering. Among them, there are a total of 9 asphalt adhesivity tests [2], corresponding to the testing of the asphalt viscosity of different viscosity units. The test methods and corresponding equipment are shown in Table 2. There is still no verification regulation for most equipment, and the testing range of viscosity is 0.0036 Pa·s to 58,000 Pa·s. The Test Specification stipulates that most of the testing equipment required for the test of multiple parameters of asphalt, such as the dynamic modulus and cohesion, needs to be calibrated with the viscosity standard under high temperature. However, such viscosity standard is monopolized by the companies such as the Cannon Company of the United States. The coverage of the system for quantity value dissemination of viscosity in China is relatively narrow, resulting in the failure in determining the technical status of in-service testing equipment.

Table 1. Test methods for asphalt adhesivity, and testing equipment condition

S/N	Test	Testing equipment	Standard specification of equipment
1	T0619-2011 Kinematic Viscosity Test of Asphalt (Capillary Method)	Capillary	JJG 154-2012 Standard Capillary Viscometers JJG 155-2016 Routine Capillary Viscometers
2	T0620-2000 Kinematic Viscosity Test of Asphalt (Vacuum Decompression Capillary Method)	Vacuum decompression capillary	JJG (JT) 172-2021 Vacuum decompression capillary viscometer of asphalt
3	T0621-1993 Standard Viscosity Test of Asphalt (Standard Viscometer Method for Road Asphalt)	Standard viscometer of road asphalt	JJG (JT) 055-2004 Verification regulation of asphalt standard viscometer
4	T0622-1993 Engler Viscosity Test of Asphalt (Engler Viscometer Method)	Engla viscometer	None
5	T0623-1993 Saybolt Viscosity Test of Asphalt (Saybolt Heavy Oil Viscometer Method)	Saybolt heavy-oil viscometer	None
6	T0624-1993 Asphalt Toughness and Tenacity Test	Toughness and tenacity tester	None
7	T0625-1993 Rotational Viscosity Test of Asphalt (Brockfield Viscometer Method)	Brockfield viscometer	JT/T 944-2014 Blockfield Viscometer of Asphalt (the verification regulation in preparation)
8	T0627-2011 Bending Creep Stiffness Test of Asphalt (Bending Beam Rheometer Method)	Bending beam rheometer	None
9	T0628-2011 Rheological Property Test of Asphalt (Dynamic Shear Rheometer)	Dynamic shear rheometer	JJG (JT) 157-2020 Dynamic shear rheometer

For example, when the metrological verification regulation of the JJG (JT) 157-2020 Dynamic Shear Rheometer was compiled, the dynamic shear rheometer of an import company of a key laboratory in a university was selected for the test, and the N2700000SP viscosity standard of Cannon Company that is specified by American standard AASHTO T315 was adopted (see Table 2 for test data) for calibration. The test result showed that the relative indication error was 9.64% to 19.95%, which was much higher than the requirement of ±3.0%. The manufacturer verified and confirmed that measurement accuracy was affected by the inaccurate temperature of the equipment (the error exceeded 10°C).

Table 2. Equipment verification data

Unit: Pa

Temperature/°C	First test value	Relative indication error	Second test value	Relative indication error	Third time Test value	Relative indication error	Mean of test values	Standard value	Relative indication error
52	6738.35	3.70%	6836.95	5.22%	7797.44	20.00%	7124.25	6498	9.64%
58	4535.2	6.06%	4595.1	7.46%	5194.46	21.48%	4774.92	4276	11.67%
64	3119.52	7.72%	3178.84	9.77%	3562.03	23.00%	3286.797	2896	13.49%
70	2247.43	12.82%	2245.44	12.72%	2514.4	26.22%	2335.757	1992	17.26%
76	1603.7	15.37%	1603.64	15.37%	1794.55	29.10%	1667.297	1390	19.95%

3. Current situation of viscosity standard in foreign countries

Table 3. Viscosity standard for NIST

Catalog Number	Viscosity Standard	Insert Table 1 Kinematic Viscosity in mm²/s (Centistokes)								
		20°C	25°C	37.78°C	40°C	50°C	60°C	80°C	98.89°C	100°C
9727-C10	N.4	0.47	0.45	0.41	0.40	—	—	—	—	—
9727-C15	N.8	0.74	0.70	0.61	0.60	—	—	—	—	—
9727-C20	N1.0	1.3	1.2	1.0	0.97	0.91	—	—	—	—
9727-C22	N2	2.9	2.6	2.1	2.0	1.7	—	—	—	—
9727-C25	S3	4.6	4.0	3.0	2.9	2.4	—	1.5	1.2	1.2
9727-C27	N4	6.7	5.8	4.2	4.0	3.2	—	1.9	1.5	1.5
9727-C30	S6	11	8.9	6.0	5.7	4.4	—	2.4	1.8	1.8
9727-C31	N7.5	14	12	8.0	7.5	5.8	—	3.1	2.3	2.3
9727-C32	N10	21	17	11	10	7.3	—	3.5	2.5	2.4
9727-C34	N14	30	25	15	14	10	—	5.0	3.5	3.4
9727-C35	S20	44	34	20	18	13	—	5.6	3.9	3.8
9727-C36	N26	57	46	27	25	18	—	7.9	5.3	5.2
9727-C37	N35	87	66	35	32	21	—	8.5	5.4	5.3
9727-C38	N44	110	86	48	44	30	—	12	7.7	7.5
9727-C40	S60	160	120	60	54	35	—	12	7.7	7.5
9727-C41	N75	200	150	82	75	50	—	19	12	12
9727-C42	N100	330	230	110	97	60	—	19	11	11
9727-C43	N140	400	300	160	140	90	—	31	19	18
9727-C45	S200	660	460	200	180	105	—	30	17	16
9727-C46	N250	770	570	280	250	160	—	51	30	29
9727-C47	N350	1300	850	350	310	170	—	46	24	23
9727-C48	N415	1400	990	470	420	250	—	77	43	41
9727-C50	S600	2400	1600	600	520	280	—	67	34	33
9727-C51	N750	2600	1900	850	750	440	—	130	68	66
9727-C52	N1000	3400	2400	—	940	550	350	150	—	80
9727-C53	N1400	5100	3600	—	1400	820	510	220	—	120
9727-C55	S2000	8300	5300	1900	1600	800	—	160	75	72
9727-C56	N2500	8400	6000	—	2500	1500	950	430	—	230
9727-C57	N4000	20000	12000	—	3400	1600	850	290	—	120
9727-C58	N5100	28000	18000	—	5100	2500	1300	420	—	170
9727-C60	S8000	41000	25000	8000	6700	3200	—	530	—	215
9727-C61	N10200	58000	36000	—	10200	4900	2500	775	—	300
9727-C62	N15000	77000	47000	—	13000	6100	3000	980	—	360
9727-C63	N18000	103000	64000	—	18000	8500	4300	1320	—	500
9727-C65	S30000	—	79000	27000	22000	11000	—	1700	—	630

Table 4. Viscosity standard for PTB

Reference liquid	Kinematic viscosity in mm²/s at					Uncertainty (%)
	20 °C	23 °C	25 °C	40 °C	100 °C	
1B	1.2	1.2	1.2	0.96	-	0.2
2B	3.7	3.5	3.3	2.5	-	0.2
5A	6.1	5.6	5.3	3.7	-	0.2
10A	12	11	10	6.4	-	0.2
20A	23	20	19	11	-	0.2
20B	36	31	28	16	3.5	0.2
50B	65	56	50	25	4.7	0.25
100A	105	91	83	44	8.1	0.25
100B	150	130	120	60	10	0.25
200A	250	210	180	79	9.6	0.25
500A	450	380	340	150	19	0.28
500B	880	700	600	230	19	0.28
1000A	1300	1100	930	390	40	0.28
2000A	2000	1600	1400	520	38	0.28
5000A	4500	3600	3200	1200	97	0.35
10000A	9000	6900	5800	1700	71	0.38
10000B	17000	13000	10000	3000	110	0.45
20000A	21000	16000	14000	4300	180	0.45
20000B	37000	28000	24000	7200	270	0.5
50000A	53000	44000	38000	15000	1300	0.5
100000A	101000	76000	63000	17000	450	0.7
100000B	143000	107000	89000	24000	620	0.8
200000B	358000	268000	222000	61000	1500	0.8
500000B	775000	580000	481000	131000	3200	1.0

As synthetic oil has a simple composition, the fixed molecular structure, narrow molecular weight distribution, almost no double bond, and good chemical and physical stability, it has been selected by the National Institute of Standards and Technology (NIST), the Physikalisch-Technische Bundesanstalt (PTB) and other measurement research institutions as the viscosity standard, as shown in Tables 3 and 4. Such viscosity standard has a long effective period. For example, the viscosity standard for NIST can be effective for about 2 years. In addition, the viscosity standard for synthetic oil has good thermal stability, and has almost no volatilization in the process of heating to 100℃, which is suitable for high temperature setting. The viscosity standard for both NIST and PTB can provide the viscosity values from 20℃ to 100℃, which is convenient for the customers in many fields.

4. Current status of existing viscosity standards in China

China's current viscosity measurement system can only transfer viscosity values in the range of (20 to 40) ℃, as the poor thermal stability of the current viscosity standards in China results in unavailability of the wide temperature range setting. In China, the current viscosity standard is mainly produced by blending two or more types of base oil. The base oil of the viscosity standard liquid with the viscosity lower than 500 mPa·s at 20°C is the petroleum product, and the base oil of the viscosity standard with the viscosity of (500 to 100,000) mPa·s is methyl silicone oil. The petroleum product is a multi-hydrocarbon mixture consisting of the straight-chain paraffin, cyclic hydrocarbon and aromatics. There are more straight-chain paraffins in a low viscosity range, while there are more cycloalkanes and aromatics in a high viscosity range. The base oil types that are used to prepare the standard liquid are kerosene, transformer oil, hydraulic oil, aeroplane oil, steam turbine oil, etc.

Table 5. List of existing viscosity standards in China

No. of viscosity standard liquid	Constant temperature (℃)	Kinematic viscosity (mPa·s)	Uncertainty (mPa·ss)
GBW(E)130624	20	5.022×10^4	0.88
	25	3.117×10^4	0.88
	30	1.974×10^4	0.88
	40	8538	0.80
	80	624.0	0.63
	100	239.9	0.63
GBW(E)130625	20	1.014×10^5	0.88
	25	6.286×10^4	0.88
	30	2.998×10^4	0.88
	40	1.718×10^4	0.88
	80	1211	0.80
	100	451.8	0.63
GBW13615(1807)	20	1.2298×10^5	0.50
GBW13615(1806)	20	5064.9	0.39
GBW13609(1706)	20	1093.7	0.33
GBW13610(1706)	20	2107.8	0.39
GBW13613(1706)	20	20492	0.44
GBW13605(1706)	20	51.859	0.21

As shown in Table 5, the maximum nominal value of the viscosity standard in China at 20℃ is 1.2298 × 105 mPa·s, which cannot meet the use requirement of the calibration of asphalt viscosity testing equipment. Similarly, as there are multiple components with different molecular weights and molecular structures in the system, the use of the viscosity standard liquid over a large temperature range is greatly restricted.

5. Developing technology of high viscosity standard

The viscosity standards are different from the chemical products for general industrial purposes. The polymerization degree and structure of polymers are multi-dispersed. The in-depth research on the synthesis conditions, equipment, processes and other aspects must be conducted to obtain low-viscosity synthesized raw oil products that have Newtonian flow properties and meet the requirements of viscosity values, that have good uniformity and can be stored stably, and that have no corrosivity but rich sources.

In the analysis of the viscosity standard for NIST, it is found that polyisobutylene (PIB) is used as the raw material for many high viscosity standards. Polyisobutylene (PIB) is a typical linear polymer produced through the cationic polymerization of the monomer isobutylene (IB)[3]. There is no asymmetric carbon atom in the structural units of PTB, and the structural units are connected in a head-to-tail sequence. Its molecular structure has the methyl side-chain, and a long-chain high polymer with the double bonds at the end. It has unique and precious properties such as relative inertness, chemical stability and excellent ozone resistance, ultraviolet resistance, corrosion resistance and electrical insulation performance[4,5]. It is widely used in lubricating oil, the raw materials of PIB series rubber, adhesives, plasticizers, softeners, electrical insulating materials, interstitial sealing materials, gasoline detergents, the matrix adhesives of chewing gum and many other aspects[6-8]. The polyisobutylene(PIB) with a low polymerization degree is generally called as the low-molecular-weight (LMW) polyisobutylene(PIB). As the viscosity standard, Polyisobutylene(PIB) has the advantages of good Newtonian properties, low toxicity, non-corrosiveness, good chemical stability (can be stored stably below 120℃), good uniformity, physical stability, etc.

In 1873, the former Soviet Бутлеров first discovered the polymerization of isobutylene under the action of BF_3, and determined its structure through detailed research. Since then, the countries such as the United States, Germany, Britain and Japan have successively carried out the research on polyisobutylene (PIB). In 1940, the German BASF company established the 6,000 t/a polyisobutylene (PIB) production plant for the first time. In 1942, Exxon of the United States established the first industrial-scale butyl rubber factory and produced polyisobutylene (PIB) products. At present, the countries such as the United States, France and Germany can produce polyisobutylene (PIB), and the continuous polymerization technology of the German BASF company and the United States Standard oil company was mainly adopted. The production process of LMW polyisobutylene (PIB) of Exxon Company in the United States is the same with it. For the production, high-purity isobutylene and isobutane or hexane are mixed, and polymerized at (-10 to -20)℃ with $AlCl_3$ or BF_3 as the catalyst. The manufacturing processes of HMW polyisobutylene (PIB) are different. The Exxon company adopts the slurry polymerization process with $AlCl_3$ as the initiator. The stability of the polymerization slurry in this process is the key technical problem affecting the continuous operation of PIB. The Cosden Company of the United States uses the mixed C_4 fraction of the petroleum refinery to synthesize polyisobutylene (PIB), and adopts the $AlCl_3$ initiation system. The 1-butylene in the raw material is a mild inhibitor, which can reduce the yield of low-polymerization isobutylene but has a little effect on the average molecular weight. 2-butene is both an inhibitor and a chain transfer agent, which can reduce the yield and average molecular weight. Therefore, the reaction process of using mixed C_4 to synthesize polyisobutylene (PIB) is the polymerization process of isobutylene in the presence of the inhibitor and chain-transfer agent[9].

In the 1980s, China carried out the relevant research. At first, the research was for the production of the barium raw material of oil detergent dispersant for internal combustion engines. The raw material for production was C_4 fraction, and the $AlCl_3$ sesquialuminium is used as the catalyst. The obtained molecular weights of the products are 1,000 to 3,000. In the early 1980s, the Lanzhou Petrochemical Refinery and Jinzhou Refinery established 500 t/a and 300 t/a production plants respectively. In the later stage, the General Plant of the Daqing Petrochemical Company added the production of the polyisobutylene (PIB) with the molecular weights of 20,000 to 40,000; the mixed C4 was adopted as the raw material, and toluene-AlCl3 as the system catalyst. All the polyisobutylene (PIB) produced by the above manufacturers in 1977 was light yellow and could not be used in white products; additionally, the products with the molecular weights above 20,000 contained diluted oil, which restricted production and development. In 1988, Research Institute of the Jilin Petrochemical Company carried out the research of colorless HMW Polyisobutylene (PIB), and completed lab tests, for the localization of the viscosity index improver (imported from Japan) added to the white oil exported from the Grease factory of the Jilin Petrochemical Company; it also developed colorless LMW Polyisobutylene (PIB), and established a 100 t/a pilot plant for low Polyisobutylene (PIB) to meet the demand for rat-catching gel in cooperation with the Dalian Rat Medicine Factory and Mitsui Disinfection Co. In 1995, the first 200 t/a colorless polyisobutylene (PIB) production plant was built in China. The main technical indexes of the product reached the international advanced level, which can completely replace the imported products and fill the gaps in China. The molecular weight range is between 30,000 and 100,000 [9].

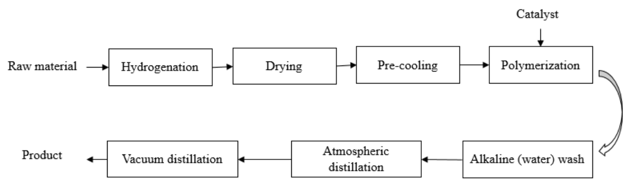

Figure 1. Synthesis process flow of highly-active polyisobutylene

The synthesis processes of high-activity polyisobutylene at home and abroad are basically the same (see Figure 1). First, the raw materials are dried, and the catalyst is added into the raw materials for polymerization; then, use the alkali to wash the polymer, and use water to wash and remove the catalyst; finally, atmospheric distillation and vacuum extraction are implemented. For the process route of relative LMW synthesis, the key technology is the synthesis catalyst system[10,11]. The most common catalysts in industry are $AlCl_3$ and BF_3 catalysts, of which the production of PIB using the AlCl3 catalyst system is the largest. Exxon company adopts the slurry polymerization process, with $AlCl_3$ as the catalyst; the isobutylene monomer concentration is 30%, and the $AlCl_3$ slurry concentration is 5%; the reaction temperature is -40°C, and the catalyst slurry is pre-cooled to -20°C before adding. In recent years, a lot of research on the highly-active PIB with a mass fraction of terminal α-double bonds greater than 70% has been carried out at home and abroad, and many patented technologies have been invented[12-21]. For example, British Petroleum Chemicals Co., Ltd. provides the BF3 complex catalyst to control the quality of PIB products in IB liquid phase polymerization. Germany BASF Company and Amoco Company respectively have provided polymerization methods, that is, in the presence of the BF_3 complex catalyst, the IB liquid polymerization is utilized to prepare the relative LMW with the mass fraction of the terminal α-double bonds greater than 80%.

The analysis of highly-active polyisobutylene products mainly includes the analysis of the active terminal content in the highly-active polyisobutylene, and the analysis of the molecular weight and molecular weight distribution of highly active polyisobutylene. The analysis methods mainly include the nuclear magnetic resonance analysis (NMR), infrared spectrum analysis (FTIR) and high-performance gel chromatography (GPC)[22,23].

6. Analysis of current research level and development trend

Table 6. Statistics of the countries participating in the CCM's second international key comparison of viscosity

Laboratory	Standard Liquid A			Standard Liquid B		
	-40°C	-20°C	20°C	20°C	100°C	150°C
Cannon	X	X	X	X	X	X
INRiM			X	X	X	X
LNE	X	X	X	X	X	X
NMi			X		X	
NMIJ/AIST	X		X	X	X	
NIM			X	X		
PTB	X	X	X	X	X	X
SMU			X	X		
VNIIM	*withdrawn*	X	X	X	X	
BEV		X	X	X	X	X
INM	X	X	X	X	X	X
INMETRO			X	X	X	
IPQ			X	X		
NIS			X	X		

* *Bold text denotes laboratory with independent viscosity scale*

The key comparison of viscosity values that is organized by the Working Group on Viscosity of the Consultative Committee for Mass and Related Quantities (CCM-WGV) is the most authoritative viscosity comparison in the world, and an important basis for measuring the viscosity measurement level of a country. Over the years, China has made great achievements in the international comparison. However, the leading laboratories have been requiring the viscosity comparison in a wide temperature range for years, with a large temperature span from - 40°C to 150°C, as shown in Table 3. Due to the poor thermal stability of the viscosity standard, the development of viscosity measurement in the wide temperature range in China has been greatly restricted. Therefore, China can only participate in the comparison of the narrow temperature range of (20°C to 40°C). This has been a bottleneck restricting the development in the professional field of viscosity in China.

As an important performance index of asphalt, the asphalt viscosity under the high temperature is of great significance to evaluate the dynamic stability of the pavement and rut resistance under the high temperature. It is a key basic index for the construction of the long-life pavement in a country with the strong transportation network. However, at present, the high viscosity standard that are used in China for verifying and testing the performance of the high-temperature asphalt viscosity equipment have not fulfilled the independent controllability of the high viscosity standard and key technologies. Therefore, the industry will pay more and more attention to the basic research on the preparation, production, customization and other aspects of the high viscosity standard. On the one hand, it can better meet the use need of the customer, provide services in production, and reduce the customer's economic pressure; On the other hand, it prepares for expanding the temperature range of viscosity measurement in China, meets the need in the field of transportation, improves the level of viscosity measurement in China, and fulfills the stability and reliability of the quantity value dissemination and traceability in the field of road engineering, which are conducive to the healthy development of the field of road engineering.

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