Production Technology of Emulsion Polymerized Styrene Butadiene Rubber (EPSBR)

Science & Technology Management Department

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CHINA NATIONAL PETROLEUM CORPORATION

CNPC is dedicated to providing EPSBR products and technical services with exquisite technology and wide experience.

Contents

China National Petroleum Corporation (CNPC) is a state-authorized investment agency and a state holding company. As an integrated oil company of cross-regions, cross-industries and cross-countries, it adopts modern enterprise system to realize the integration of upstream and downstream operations, internal and external trade and production and marketing. CNPC has 17 upstream companies, 33 downstream companies and 36 large-scale marketing companies. It is China's largest producer and supplier of oil and gas, and also one of the largest refined oil products and petrochemicals. In 2010 CNPC produced 105 million tons of c rude oil and 72.5 billion cubic meters of natural gas, while c rude processing volume reached 135 million tons. The total revenue of RMB 1,720 billion with a profit of RMB 172.7 billion had been achieved the same year. Its profit is among the highest of the domestic enterprises in China.

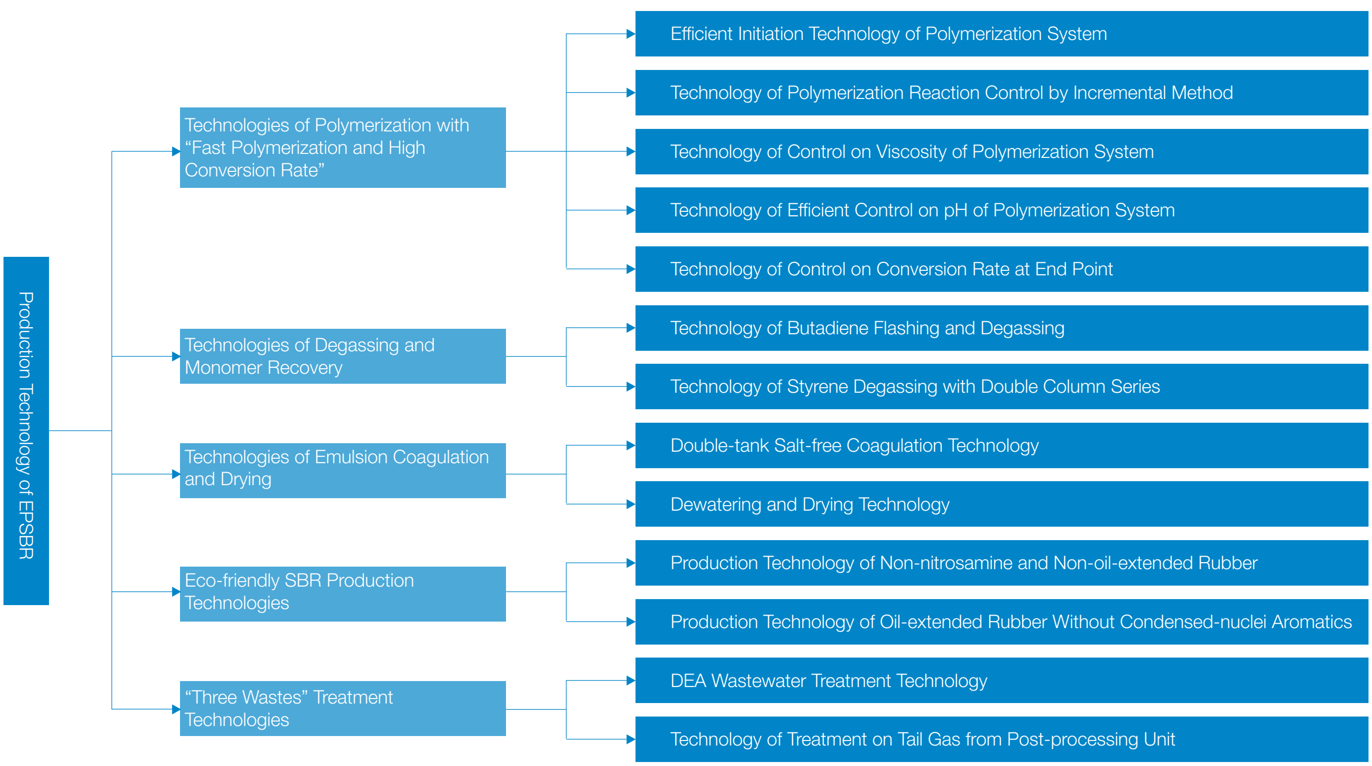
CNPC was ranked 10th in Fortune Global 500 in 2010 and 5th among global top 50 oil companies. CNPC strictly follows by t he combined strategies of increasing resource capacity, expanding market shares and consolidating the international role, and persists in regarding technical innovation as a key framework to advance technological progress. To develop its core businesses, focuses will be placed on the solutions of key bottleneck technologies and key proprietary technologies. Thanks to continuously improving of the technical innovation system, optimizing the configuration of technological resources and strengthening the construction of strong talent teams, CNPC's technological creativity has been considerably upgraded. Consequently, a large number of technologies have been developed independently, with its own intellectual property.

The Production Technology of EPSBR is one of representatives for major innovations of CNPC.

1 Introduction

China National Petroleum Corporation (CNPC) is a state-authorized investment agency and a state holding company. As an integrated oil company of cross-regions, cross-industries and cross-countries, it adopts modern enterprise system to realize the integration of upstream and downstream operations, internal and external trade and production and marketing. CNPC has 17 upstream companies, 33 downstream companies and 36 large-scale marketing companies. It is China's largest producer and supplier of oil and gas, and also one of the largest refined oil products and petrochemicals. In 2009 CNPC produced 103 million tons of c rude oil and 6 8.3 billion cubic meters of natural gas, while c rude processing volume reached 125 million tons. The total revenue of RMB 1,220 billion with a profit of RMB 125.2 billion had been achieved the same year. Its profit is among the highest of the domestic enterprises in China.

CNPC was ranked 13th in Fortune Global 500 in 2009 and 5th among global top 50 oil companies. CNPC strictly follows by t he combined strategies of increasing resource capacity, expanding market shares



and consolidating the international role, and persists in regarding technical innovation as a key framework to advance technological progress. To develop its core businesses, focuses will be placed on the solutions of key bottleneck technologies and key proprietary technologies. Thanks to continuously improving of the technical innovation system, optimizing the configuration of technological resources and strengthening the construction of strong talent teams, CNPC's technological creativity has been considerably upgraded. Consequently, a large number of technologies have been developed independently, with its own intellectual property.

The Production Technology of EPSBR is one of representatives for major innovations of CNPC.

Since 1960, CNPC has been dedicated to the production and research of EPSBR and nowadays stands as a major emulsion polymerized styrene butadiene rubber (EPSBR) manufacturer and leading research force with exquisite experience and abundant achievements in production technology and development of new EPSBR products. CNPC's independent innovation yields the unique technology of producing EPSBR at low temperature with "fast polymerization and high conversion rate", leading the domestic market.

CNPC's Production Technology of EPSBR is characterized by short reaction time, high conversion rate of polymerization, low viscosity of system, high polymerization stability, low consumption of additives, low gel content and high product performance. Moreover, features, such as mature techniques, stable production and operation, high single line capacity, long operation cycle, advanced process control, safety and environmental friendliness, ensure the safe, stable, long-periodic, full-load and optimum operation of the equipment.

With high overall performance and quality of advanced international standards, SBR produced by Emulsion Polymerization Technology at low temperature with "fast polymerization and high conversion rate" has been introduced to markets at home and abroad, being widely used for various rubber products such as tire, footware, automobile parts, rubber tubes, conveyer belt.

Application of this technology has promoted the annual production capacity of EPSBR of CNPC Jilin Petrochemical Company from 90,000t to 140,000t, and CNPC Lanzhou Petrochemical Company from 55,000t to 155,000t, in addition to which, "200,000t/an SBR equipment" of CNPC Fushun Petrochemical Company is under construction.

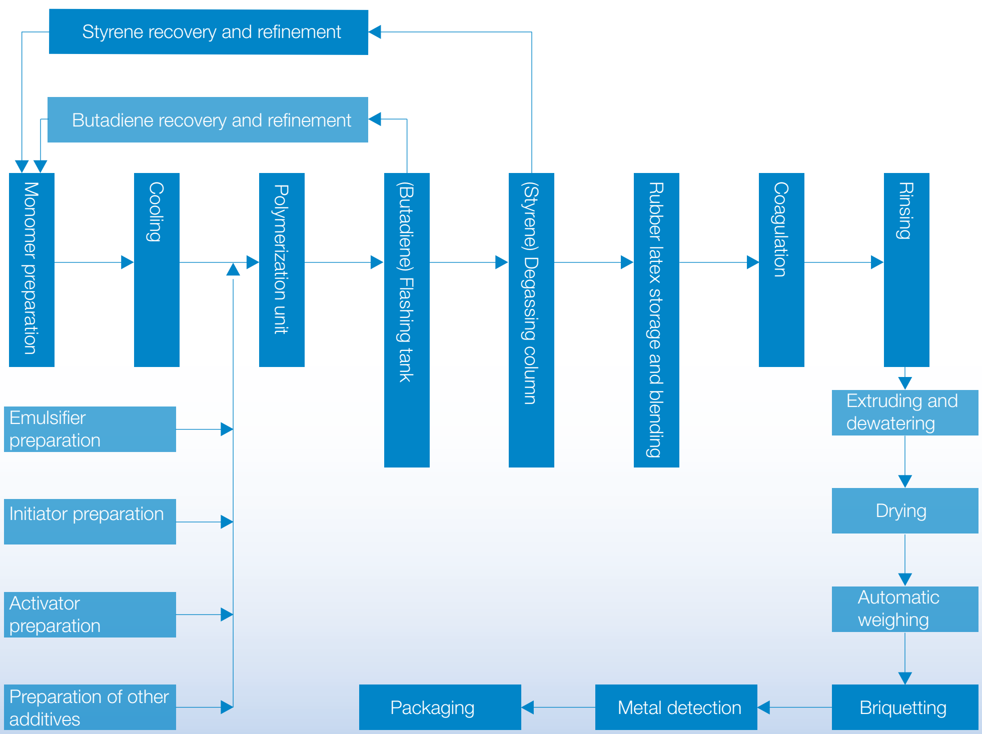
The current annual production of EPSBR by CNPC is 300,000t, taking 40% of China's market share with an annual production value of RMB 5 billion and benefit of RMB 600 million. After the SBR equipment of CNPC Fushun Petrochemical Company goes into operation, the annual production capacity of EPSBR by CNPC will get up to 500,000t, taking 50% of China's market share with an annual production value of RMB 8 billion and benefit of RMB 1 billion.

2 Unique Technologies

Production Technology of EPSBR

EPSBR is general-purpose synthetic rubber produced by low temperature emulsion polymerization of butadiene and styrene, which is applicable to tire, rubber tubes, conveyer belt and rubber overshoes due to its fine elasticity, high strength, high abrasive resistance, anti-cracking property and excellent wet traction.

EPSBR production equipment consists of six process units—monomer storage and preparation unit, chemical preparation unit, polymerization unit, monomer recovery unit, rubber latex storage and blending unit as well as coagulation, drying & package unit.



Process Flow in EPSBR Equipment

CNPC's EPSBR production technological innovation adopts the feeding process with the incremental method of molecular weight regulator and emulsifier, which brings about more stable polymerization system, more uniform quality and lower additive consumption. The rate of premium-grade product is improved from 98.5% to 99.8%, and the consumption of molecular weight regulator per unit product is reduced by 9% while emulsifier by 3%.

The system viscosity control at the later stage of polymerization reaction is one of the critical factors that ensure the heat transfer effect and long-periodic stable operation of the equipment. Production Technology of EPSBR covers the innovated viscosity control technique of polymerization system to reduce the system viscosity at the later reaction stage from 300 cp to 150 cp, to improve the heat transfer effect significantly, to decrease the rubber sticking to tank reactor wall evidently and to lengthen the operation cycle from one year to over three years.

The use of eco-friendly additives and extender oil upgrades the leading product quality which has reached the international premium standard and conforms to the European environmental standards. SBR 1500E and SBR 1502E do not contain the carcinogen, nitrosamine; SBR 1723N and SBR 1739N do not contain nitrosamine while their condensed-nuclei aromatics contents conform to the EU standards. Therefore, the pollution to the environment is decreased and harm to human health lessened.

Technologies of Polymerization with "Fast Polymerization and High Conversion Rate"

As per polymerization kinetics, "fast polymerization and low conversion rate" and "slow polymerization and high conversion rate" are two currently typical techniques for EPSBR production.

Through its unremitting exploration and innovation, CNPC has developed the technique of "fast polymerization and high conversion rate" for EPSBR production, which breaks the restriction brought by the conflict between the fast polymerization and high conversion rate. This technique combines the advantages of the two above-mentioned techniques, thus obtaining the proper bond of fast polymerization and high conversion rate. With this technique, the conversion rate of polymerization gets up to 70%±2% and the polymerization time is only 10h. This technique promotes the level of EPSBR technology and enriches the SBR Emulsion Polymerization Technology, thus allowing the existence of three types of techniques for EPSBR production—"fast polymerization and low conversion rate", "slow polymerization and high conversion rate" and "fast polymerization and high conversion rate".

1. Efficient Initiation Technology of Polymerization System

The EPSBR Production Technology with “Fast Polymerization and High Conversion Rate” utilizes the efficient initiation system featured by the simple initiator preparation process, high initiation efficiency and low oxidizer consumption. As compared with that of the similar technology, the rate of oxidizer consumption herein is 15% lower.

2. Technology of Polymerization Control by Incremental Method

The old EPSBR production technology with “fast polymerization and low conversion rate” almost adopts the all-in process (feeding once), with simple polymerization formula and process, quite rough polymerization control, low system stability and high additive consumption.

☆ Technology of Control on Molecular Chain Structure

The feeding process characterized by the feeding molecular weight regulator with the incremental method during EPSBR production can control the EPSBR Mooney viscosity and gel content, relative molecular mass and distribution to bring about more precise control on the polymer molecular chain, lower gel content and more uniform quality.

The additive consumption is significantly lowered: the consumption rate of the molecular weight regulator charged with the incremental method is reduced by 9% as compared with that of the similar technology with the all-in process (feeding once).

☆ Technology of Polymerization Rate Increase

The feeding process characterized by the charging emulsifier with the incremental

method during EPSBR production increases the polymerization rate at the later reaction stage and the latex stability, thus generating higher SBR polymerization rate, more stable system and lower additive consumption.

☆ Technology of Control on System Viscosity During Polymerization Process

Water is supplemented during the EPSBR polymerization process to control the system viscosity, which keeps the system in low viscosity at the later reaction stage. Compared with the similar technology, this technology allows better heat transfer in the end tank, lower tank temperature, less rubber sticking and much longer operation cycle.

3. Technology of Efficient Control on pH of Polymerization System

The electrolyte system for SBR emulsion polymerization has been successfully developed. By using potassium phosphate as the electrolyte, the system does not only provide highly efficient pH buffering but has better polymerization stability. In contrast, its similar technology, taking potassium chloride as the electrolyte, does not provide pH buffering due to the lack of such function in muriate.

4. Technology of Control on Conversion Rate at End Point

The conversion rate of SBR polymerization at the end point is controlled through the control of the retention time, thus generating more precise control of the polymerization conversion rate and more uniform product quality. However, its similar technology adopts the method of controlling the polymerization conversion rate at the end point by regulating the polymerization formula, where the polymerization conversion rate at the end point is volatile, and the solid content in the product and Mooney viscosity of the raw rubber fluctuate within a wide range.

Technologies of Degassing and Monomer Recovery

1. Technology of Butadiene Flashing

CNPC employs the two-stage flashing process—pressure flashing and vacuum flashing—to remove butadiene, and use the efficient gas-phase polymerization inhibitor to prevent the formation of end polymer. This process allows rapid butadiene flashing, high removal efficiency and long operation cycle.

2. Technology of Degassing with Double Column Series

Styrene is degassed through the new-type degassing column (the first column tray and the last one are large-bore sieve trays) and double column series with excellent degassing effect, which can significantly lower the styrene content in the degassed latex (the residual styrene content is less than 300ppm). Meanwhile, the operation cycle is prolonged and the times for cleaning reduced. In the similar technology, styrene is removed by the single column process. Due to the structural problem of the first and last trays, the residual styrene content is rather high (approx. 1000ppm) with severe rubber sticking to the degassing columns, short operation cycle and frequent cleaning.

Technologies of Emulsion Coagulation and Drying

1. Double-tank Salt-free Coagulation Technology

CNPC innovated the coagulation process which applies double tanks, polymer coagulant and concentrated sulfuric acid. By comparison with its similar technology—the coagulation process by using single tank, salt and diluted acid, this process features full coagulation of colloidal particles, uniform particle size, less scrap rubber and less operation troubles of the post-processing unit.

2. Dewatering and Drying Technology

The drying oven with the single-layer structure, used for drying wet rubber and adopting the heated air circulation drying process during EPSBR production, improves the drying effect, reduce the steam consumption and lessen the troubles of the drying oven.

The pressure regulator of dehydrator enables remote automatic control and regulation of the operating room so that the level of automatic control is improved. Since the operating current of dehydrator is decreased, the On/Off pressure regulator starts automatically, which significantly drops the working load of the extruding rotor and deceleration system so that the equipment's service life is prolonged and maintenance & production costs reduced. Furthermore, the optimization of production run and the upgrading of drying oven have led to easier troubleshooting, safer operation, less circulating fans and decreased steam consumption, which increases the post-processing production load and prolongs the continuous operation period.

Eco-friendly SBR Production Technologies

1. Production Technology of Non-nitrosamine and Non-oil-extended Rubber

The eco-friendly SBR does not contain carcinogen—N-nitroso compound (nitrosamine) or its precursors. CNPC has innovated and utilizes the new-type eco-friendly terminator and eco-friendly aging resister to produce the non-nitrosamine non-oil-extended rubber, which upgrades the leading products SBR 1500 and SBR 1502 to SBR 1500E and SBR 1502E. The quality of the upgraded products reaches that of the premium products of national standard with the environmental protection indexes conforming to the European environmental standards.

In contrast, N-diethylhydroxylamine and sodium dimethyl dithio carbamate contained in the polymerization terminator by the similar technology are inclined to form secondary amine in the acid environment during latex coagulation, and secondary amine can react with nitrosation agents (e.g. sodium nitrite) and nitrogen oxides in the air, which will produce carcinogens, e.g. N-nitrosodimethylamine.

2. Production Technology of Oil-extended Rubber Without Condensed-nuclei Aromatics

Filling a large amount of petroleum distillate (instead of high molecular weight fraction in high polymer) into SBR can not only retain the mechanical property of SBR but also improve its processing characteristics.

Compared with the non-oil-extended SBR, the oil-extended SBR features good processing characteristics, low heat build-up, long flex life at low temperature, excellent traction performance and wearability—especially when used as the tread rubber. In addition, the oil-extended rubber has better plasticity, allows easy mixing and helps to increase the capacity of the rubber processing equipment. Meanwhile, the use of cheap petroleum distillate lowers the cost and improves production.

The use of the common high aromatic oil-extended SBR is increasingly restricted because its extender oil contains carcinogens—condensed-nuclei aromatics (e.g. anthracene and phenanthrene). Moreover, the eco-friendly oil-extended SBR with condensed-nuclei aromatics meeting environmental protection requirements is increasingly encouraged.

After the successful development of the eco-friendly rubber extender oil, CNPC also developed eco-friendly technology of oil-extended SBR and new products—SBR 1723N and SBR 1739N, which resolves the problems that carcinogenic condensed-nuclei aromatics (e.g. anthracene, phenanthrene) contained in the traditional extender oil release substances harmful to humans and environment during the manufacture and use of tires.

“Three Wastes” Treatment Technologies

1. DEA Wastewater Treatment Technology

CNPC independently developed the DEA processing technology for treating the wastewater in the EPSBR production process, and the water quality after the treatment with this technology conforms to Grade I discharge standard of GB8968-1996. With the new technique of “multiple efficient composite coagulant and modified circular clarifier (DEA reactor)” and after the physical and chemical treatment of wastewater, the water out of the DEA reactor is treated by the hydrolysis acidification and contact oxidation process. This technology is characterized by more reduction of COD ratio during the physical and chemical treatment, less space occupied by the wastewater treatment equipment, small investment and low treatment cost.

2. Drying Oven Tail Gas Treatment Technology

☆ Treatment by Alkali Absorption

Alkali absorption is used to treat the tail gas from the drying oven and escaped gas during the coagulation process. These gases contain styrene that is not fully separated and small molecules produced by chemical degradation, whose pollution to the environment will be reduced by alkali absorption.

☆ Heat Regenerative Waste Gas Treatment Technology

The innovated catalytic combustion is used for the treatment of tail gas from the post-processing drying oven and escaped waste gas during the coagulation process of SBR. The post-processing tail gas treatment consists of pre-treatment and combustion—the former is mainly used for removing the moisture and dust in the tail gas. The pre-treated gas should undergo the catalytic combustion at 300°C (300~350°C, at most 400 °C), where the exothermic reaction releases heat used for pre-heating the inlet gas. The gas after combustion and heat transfer will be emitted through the 25m-high chimney. The final products of the treated gas are CO_2 and water, which are favorable to both the cleaner production and the environment.

3 Application Cases

CNPC has established a SBR production line of 50,000 t/a in CNPC Jilin Petrochemical Company and technically upgraded two original SBR production lines, thus promoting the production capacity of CNPC Jilin Petrochemical Company from 90,000 t/a to 140,000 t/a with the output of over 160,000 t/a;

By adopting the Production Technology of EPSBR, CNPC has established the SBR production equipment of 100,000 t/a and technically upgraded the original SBR production equipment of 55,000 t/a, thus enabling the SBR production capacity of CNPC Lanzhou Petrochemical Company to reach 155,000 t/a.

Now CNPC is establishing “SBR equipment of 200,000 t/a” for CNPC Fushun Petrochemical Company with the Production Technology of EPSBR.

4 R&D Equipment

The flask-shaking batch apparatus for emulsion polymerization and the intermittent trial apparatus for emulsion polymerization are developed independently by CNPC. The flask-shaking batch apparatus for emulsion polymerization can perform parallel experiments of 16 flasks simultaneously, which is fit for developing the low temperature EPSBR formula. The intermittent trial apparatus for emulsion polymerization includes four systems—butadiene refinement system, polymerization system, degassing system and coagulation system. The overall system controlled by Cs (computers) is highly automated with precise control, stable operation, safe and reliable system, easy and convenient operation and maintenance. In addition, the parameter data, such as temperature, pressure and flow, can be monitored, controlled, collected, stored, recorded and printed. The system is applicable to the development of the low temperature EPSBR polymerization formula and process.

5 Qualification and Standard

Qualification

Standard

Among the total 61 standards, there are ten for SBR 1500 (E), ten for SBR 1502 (E), ten for SBR 1712, eleven for SBR 1778, ten for SBR 1723N and ten for SBR 1739N.

SBR1500 (E) Product Standard

No.	Indexes		Premium grade	Grade I	Qualified
1	Mass fraction of volatile,%		≤ 0.60	≤ 0.80	≤ 1.00
2	Mass fraction of ash content,%		≤ 0.50	≤ 0.50	≤ 0.50
3	Mass fraction of organic acid,%		5.00 ~ 7.25	5.00 ~ 7.25	5.00 ~ 7.25
4	Mass fraction of soap, %		≤ 0.50	≤ 0.50	≤ 0.50
5	Mass fraction of bound styrene, %		22.5 ~ 24.5	22.5 ~ 24.5	22.5 ~ 24.5
6	Mooney viscosity of raw rubber, $ML_{1+4}^{100^{\circ}C}$		47 ~ 57	46 ~ 58	45 ~ 59
7	Mooney viscosity of M-PUR, $ML_{1+4}^{100^{\circ}C}$		≤ 88	≤ 88	≤ 88
8	300% tensile stress at a given elongation (145℃), MPa	25min	11.8 ~ 16.2	10.7 ~ 16.3	——
		35min	15.5 ~ 19.5	14.4 ~ 20.0	14.2 ~ 20.2
		50min	17.3 ~ 21.3	16.2 ~ 21.8	——
9	Tensile strength (145℃ ×35min), MPa		≥ 24.0	≥ 23.0	≥ 23.0
10	Elongation at break (145℃ ×35min), %		≥ 400	≥ 400	≥ 400

SBR1502 (E) Product Standard

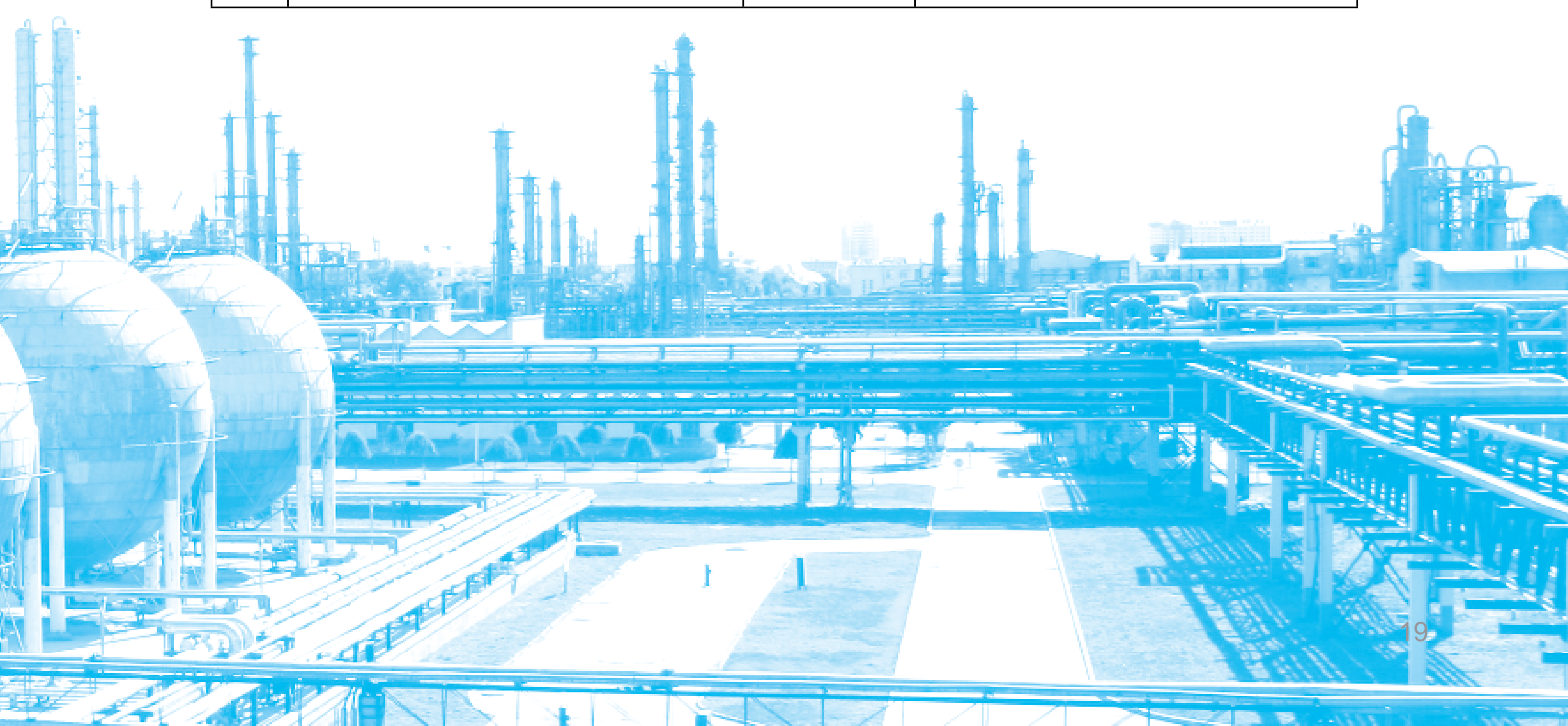
No.	Indexes		Premium grade	Grade I	Qualified
1	Mass fraction of volatile, %		≤ 0.60	≤ 0.75	≤ 0.90
2	Mass fraction of ash content,%		≤ 0.50	≤ 0.50	≤ 0.50
3	Mass fraction of organic acid,%		4.50 ~ 6.75	4.50 ~ 6.75	4.50 ~ 6.75
4	Mass fraction of soap,%		≤ 0.50	≤ 0.50	≤ 0.50
5	Mass fraction of bound styrene,%		22.5 ~ 24.5	22.5 ~ 24.5	22.5 ~ 24.5
6	Mooney viscosity of raw rubber, $ML_{1+4}^{100℃}$		45 ~ 55	44 ~ 56	44 ~ 56
7	Mooney viscosity of M-PUR, $ML_{1+4}^{100℃}$		≤ 90	≤ 90	≤ 90
8	300% tensile stress at a given elongation (145℃ ×), MPa	25min	15.7 ~ 19.7	15.2 ~ 20.2	15.2 ~ 20.2
		35min	18.6 ~ 22.6	18.1 ~ 23.1	18.1 ~ 23.1
		50min	19.5 ~ 23.5	19.0 ~ 14.0	19.0 ~ 14.0
9	Tensile strength (145℃ ×35min), MPa		≥ 25.5	≥ 24.5	≥ 24.5
10	Elongation at break (145℃ ×35min), %		≥ 340	≥ 330	≥ 330

SBR1712 Product Standard

No.	Indexes		Premium grade	Grade I	Qualified
1	Volatile, %		⩽ 0.6	⩽ 0.8	⩽ 1.0
2	Total ash, %		⩽ 0.50		
3	Organic acid, %		3.9 ~ 5.7		
4	Soap content, %		⩽ 0.50		
5	Bound styrene, %		22.5 ~ 24.5		
6	Oil content, %		25.3 ~ 29.3	24.3 ~ 30.3	
7	Mooney viscosity of raw rubber, $ML_{1+4}^{100^\circ C}$		46 ~ 54	43 ~ 55	42 ~ 56
8	Mooney viscosity of M-PUR, $ML_{1+4}^{100^\circ C}$		⩽ 70		
9	300% tensile stress at a given elongation (145℃ ×), MPa	25min	9.8 ~ 13.8	9.3 ~ 14.3	
		35min	12.1 ~ 16.1	11.6 ~ 16.6	
		50min	13.0 ~ 17.0	12.5 ~ 17.5	
10	Tensile strength (145℃ ×35min), MPa		⩾ 19.4	⩾ 18.4	
11	Elongation at break (145℃ ×35min), %		⩾ 380	⩾ 370	

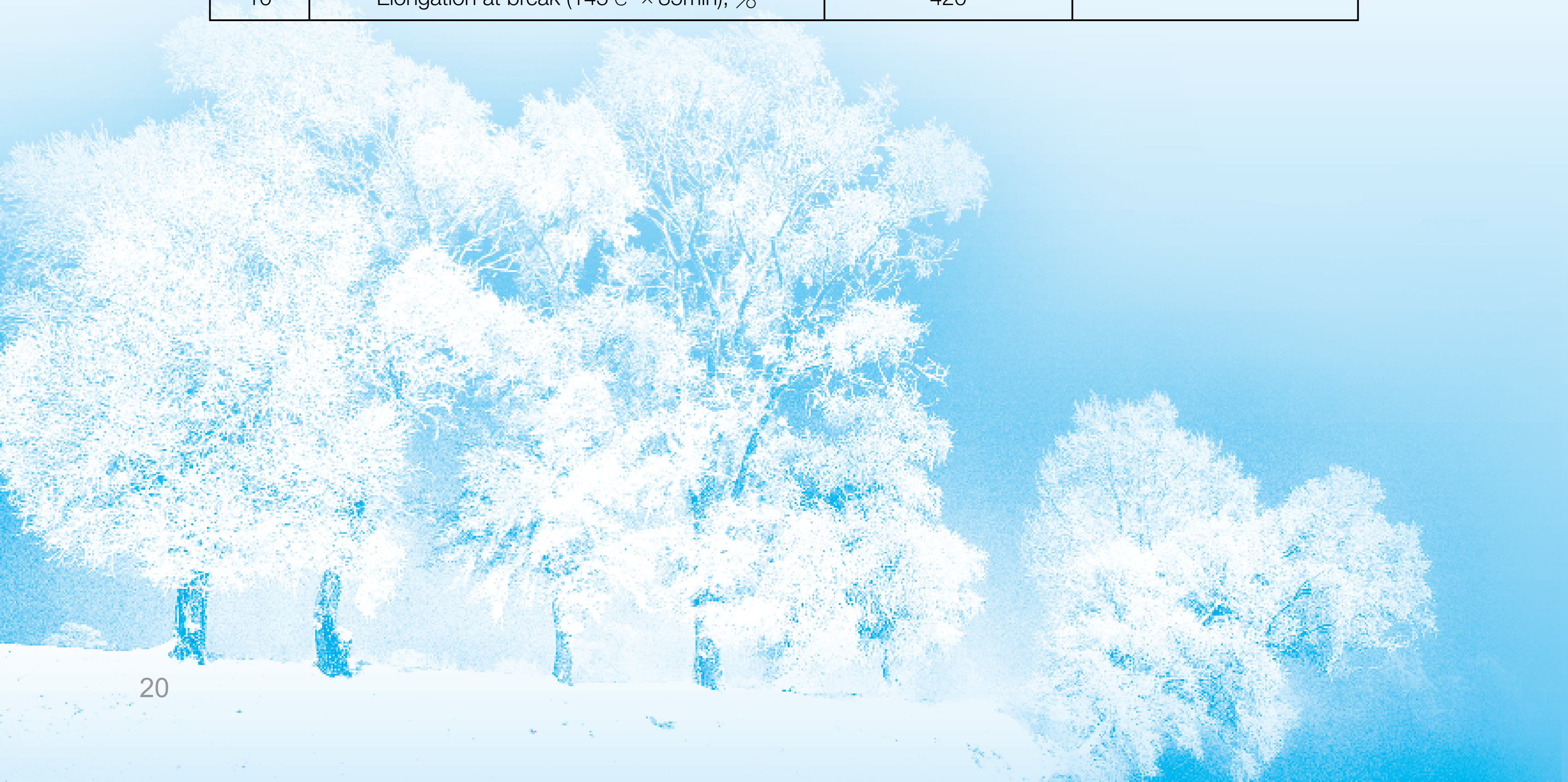
SBR1778 Product Standard

No.	Indexes		Premium grade	Grade I	Qualified
1	Volatile, %		≤ 0.75	≤ 1.0	
2	Total ash, %		≤ 0.50	≤ 1.0	
3	Organic acid, %		3.9 ~ 5.7	3.9 ~ 6.8	
4	Soap content, %		≤ 0.5		
5	Bound styrene, %		22.5 ~ 24.5		
6	Oil content, %		25.3 ~ 29.3	24.3 ~ 30.3	
7	Mooney viscosity of raw rubber, $ML_{1+4}^{100℃}$		40 ~ 50	39 ~ 51	38 ~ 52
8	Mooney viscosity of M-PUR, $ML_{1+4}^{100℃}$		≤ 64		
9	300% tensile stress at a given elongation (145 ℃), MPa	25min	6 ~ 10	5 ~ 10	
		35min	10 ~ 14	9 ~ 14	9 ~ 14
		50min	12 ~ 16	11 ~ 16	
10	Mooney viscosity of M-PUR, $ML_{1+4}^{100℃}$		≥ 17.5		≥ 16.5
11	Mooney viscosity of M-PUR, $ML_{1+4}^{100℃}$		≥ 430	≥ 410	



SBR1723N Product Standard

No.	Indexes		Min.	Max.
1	Bound styrene, %		22.5	24.5
2	Volatile, %		-	0.50
3	Ash, %		-	0.40
4	Soap content, %		-	0.50
5	Organic acid, %		3.90	5.70
6	Oil content, %		25.3	29.3
7	Mooney viscosity of raw rubber, $ML_{1+4}^{100℃}$		40	50
8	300% tensile stress at a given elongation (145℃ ×), MPa	25min	7.4	11.3
		35min	8.8	12.7
		50min	10.3	14.2
9	Tensile strength (145℃ ×35min), MPa		17.6 (180)	-
10	Elongation at break (145℃ ×35min), %		420	-



SBR1739N Product Standard

No.	Indexes		Min.	Max.
1	Bound styrene, %		38.5	41.5
2	Volatile, %		-	0.50
3	Ash, %		-	0.40
4	Soap content, %		-	0.50
5	Organic acid, %		3.90	5.70
6	Oil content, %		25.3	29.3
7	Mooney viscosity of raw rubber, $ML_{1+4}^{100℃}$		47	57
8	300% tensile stress at a given elongation (145℃ ×), MPa	25min	7.4	11.3
		35min	9.3	13.2
		50min	10.8	14.7
9	Tensile strength (145℃ ×35min), MPa		18.6 (190)	-
10	Elongation at break (145℃ ×35min), %		420	-

6 Expert Team

Li Chongjie

Professor and senior engineer. He has 25-year experience in technology and production management.
Email: jh_lcj@petrochina.com.cn

Lu Shulai

Professor and senior engineer. He has 20-year experience in the emulsion polymerization scientific research and technological development. He undertook the post-doctoral research at POLYMAT institute (Spain). Furthermore, he has won 3 provincial and ministerial-level prizes, obtained 5 national patents and published 45 academic papers.
Email: jh_lusl@petrochina.com.cn

Li Tie

Professor and senior engineer. He has 20-year experience in technology and production management.
Email: jh_litie@petrochina.com.cn

Sui Jun

Senior engineer. He has 18-year experience in EPSBR technology and production management.
Email: jh_sujun@petrochina.com.cn

Zhang Guanying
Senior engineer. He has 13-year experience in EPSBR technology and production management.
Email: jh_zhanggy@petrochina.com.cn

Liu Jiping
Senior engineer. He has 20-year experience in EPSBR technology and production management.
Email: liujip@petrochina.com.cn

He Chengquan
Senior engineer. He has 20-year experience in EPSBR technology and production management.
Email: hechengquan@petrochina.com.cn

Gong Guangbi
Ph.D., professor and senior engineer, CNPC senior technical expert. He enjoys the Special Allowance of the State Council of China. He has long been engaged in the development of synthetic rubber. Moreover, he has applied for 20 patents for invention and published over 10 academic papers as well as 5 monographs co-authored by him.
Email: gongguangbi@petrochina.com.cn

联系人：刁顺 先生
电　话：59986059
Email: sdiao@cnpc.com.cn

Contact: Mr. Diao Shun
Tel: 59986059
Email: sdiao@cnpc.com.cn