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MOLECULAR-WEIGHT DISTRIBUTION OF C9 HYDROCARBON RESINS

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This study examines the molecular-weight distribution of hydrocarbon resins obtained by oligomerization of the C9 fraction derived from liquid by-products of diesel fuel pyrolysis. Resins produced by oil-in-water and water-in-oil emulsion oligomerization, suspension oligomerization, and high-temperature post-oligomerization were fractionated using partial precipitation with benzene–ethanol mixtures. Molecular weights of the samples were determined by the cryoscopic method. The number-average, weight-average, and z-average molecular weights, the polydispersity index, and entropy-based metrics were calculated. Low-temperature emulsion and suspension processes yielded narrow distributions with low polydispersity (1.01–1.13), moderate entropy (0.80–1.08) and effective fraction numbers of 2.2–3.0. Increasing the proportion of the C9 dispersed phase in emulsions led to higher molecular-weight averages, indicating enhanced chain growth in larger organic droplets. Cyclopentadiene resins formed under high-temperature conditions exhibited higher molecular weights (630–655) and broader profiles. The styrene–cyclopentadiene resin obtained at 453 K showed the widest distribution and the strongest high-molecular-weight tail. The results demonstrate that the oligomerization route and temperature critically determine the structural uniformity of C9 resins, with low-temperature dispersion methods producing more homogeneous materials and high-temperature radical processes generating broader, heavy-tailed distributions.

Keywords: fractionation, partial precipitation, molecular weight distribution, C9 resin, oligomer.

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Introduction

Hydrocarbon resins, also called petroleum polymer resins (PRs), are oligomeric materials derived from petrochemical feedstocks. They are synthesized through the oligomerization of unsaturated hydrocarbons contained in the liquid by-products of gasoline and diesel pyrolysis – primarily the C9 fraction (422–473 K) [1,2].

The composition of these resins depends on the feedstock characteristics as well as the pyrolysis and oligomerization conditions. Hydrocarbon resins are widely applied in anti-corrosive coatings, varnishes, offset inks, and rubber compounding [1].

Hydrocarbon resins are produced by free-radical oligomerization of hydrocarbon fractions, in which the initiators are soluble in the hydrocarbon feedstock. A wide range of initiators can be used, including acyl peroxides, hydroperoxides, heat-stable alkyl peroxides, amino peroxides, and organosilicon peroxides. The process is characterized by a long oligomerization duration (6–8 h) at elevated temperatures (453–473 K). Product isolation is also challenging and typically requires both atmospheric and vacuum distillation [3].

The temperature regime is a decisive parameter in shaping the molecular-weight characteristics of resins obtained from the C9 fraction. An increase in temperature leads to a decrease in the average molecular weight due to intensified chain-transfer reactions that reduce the degree of polymerization. Conversely, lower temperatures promote the formation of resins with higher weight-average molecular weight values and, and correspondingly higher softening points [2].

To reduce the energy consumption of hydrocarbon resin production, low-temperature dispersion methods, emulsion and suspension oligomerization, have been proposed [3]. However, the C9 fraction of pyrolysis diesel fuel typically contains up to 20% dicyclopentadiene (DCPD), which does not polymerize under low-temperature radical conditions. The reactive monomer, cyclopentadiene, is formed at elevated temperatures (≈453 K) via the retro–Diels–Alder reaction [4].

For this reason, a two-stage process has been proposed in the literature, where low-temperature (303–353 K) dispersion oligomerization is complemented by a high-temperature pre-oligomerization step (453–473 K) to convert the unreacted DCPD-rich components. At the first stage, the oligomer contains mainly styrene and its derivative units typical for the C9 fraction. At the second stage, the product consists predominantly of cyclopentadiene-based oligomers, along with minor amounts of indene and styrene [3].

We assume that low-temperature dispersion oligomerization promotes the formation of hydrocarbon resins with a narrow molecular-weight distribution. Under such conditions, reduced chain-transfer and termination rates ensure more uniform chain growth, which is typical of emulsion and suspension radical polymerization systems.

In previous work [5], it was shown that the rheological behavior of hydrocarbon resins is highly sensitive to the synthesis method and temperature, reflecting differences in molecular structure and intermolecular interactions. These findings highlight the importance of studying molecular-weight distribution as a key factor governing the flow behavior and structural uniformity of hydrocarbon resins.

The study [6] compared the cooligomerization of dominant monomers of the C9 fraction under heterogeneous catalytic, homogeneous radical, and purely thermal conditions. It was shown that radical oligomerization yields the narrowest molecular-weight distributions, whereas high-temperature thermal conversion produces broad, heavy-tailed profiles with elevated weight-average and z-average molecular weights. Heterogeneous catalysis provides high resin yields but results in darker products and requires post-reaction purification.

The influence of molecular weight on the solubility of poly(ethylene succinate) polyester has been demonstrated, with higher-molecular-weight samples exhibiting lower solubility because of stronger intermolecular interactions and increased crystallinity. Solubility and fractionation behavior were assessed using a dimethyl sulfoxide–water solvent–precipitant system. The partial precipitation technique enabled the correlation between polyester solubility and its molecular-weight characteristics to be established [7].

Control of the molecular weight distribution is a key factor in achieving stable rheological and mechanical properties of polymers. Changing the reaction conditions, particularly the temperature, duration and monomer feed rate, allows for the targeted formation of the desired molecular weight distribution. The molecular-weight distribution is highly sensitive to kinetic factors and the balance of elementary steps [8,9].

An optimal, relatively narrow molecular weight distribution promotes the formation of stable oligomeric structures, whereas an excessively broad distribution leads to a deterioration in product homogeneity [10].

In oligomeric systems, the product composition and molecular-weight distribution are governed by the balance between chain-growth and chain-termination reactions. Computational modeling shows that product distributions can be predicted from kinetic parameters, highlighting the strong sensitivity of the molecular-weight distribution to the mechanism and conditions of oligomerization [11].

The efficiencies of the elution and precipitation chromatography methods were compared for the fractionation of polystyrene. Precipitation chromatography using the tetrahydrofuran–n-hexane system provided a narrower molecular weight distribution and higher fractionation selectivity than the elution method [12].

In the study [13], the reliability and reproducibility of the fractional precipitation method for determining the molecular weight distribution of polystyrene were investigated. It was established that maintaining a constant solvent-to-precipitant ratio and temperature ensures self-consistent results. The experimental data showed good agreement with gel permeation chromatography results, confirming that the fractional precipitation method can be effectively applied to evaluate the molecular weight characteristics of oligomeric and resin-like systems.

The analysis of the molecular-weight distribution of oligomers makes it possible to assess the degree of polymerization, product uniformity, and the influence of synthesis conditions on the material's properties.

The aim of this work is to investigate the molecular weight distribution of hydrocarbon resins obtained from the oligomerization of the C9 fraction and to establish the influence of synthesis conditions on the molecular-mass characteristics of the resulting resins.

Experimental

The research was conducted using a variety of hydrocarbon resins, obtained through different methods (Table 1).

The chemical composition of the obtained resins varies significantly depending on the oligomerization method and reaction temperature.

Resins **I–IX**, synthesized via low-temperature emulsion and suspension oligomerization, predominantly contain structural units derived from styrene and its substituted derivatives.

Resins **X** and **XI** were produced from a C9 fraction depleted of styrene and its derivatives, and therefore represent cyclopentadiene-based resins.

Resin **XII**, obtained through high-temperature free-radical oligomerization, is a styrene–cyclopentadiene cooligomer.

The molecular-weight distribution of the hydrocarbon resins was determined using the partial precipitation technique. Resin solutions prepared in benzene were fractionated by the incremental addition of ethanol as a nonsolvent at 293–298 K. After each addition, the precipitated fraction was isolated, dried to constant mass, and weighed. The molecular weight of each fraction was determined by cryoscopic measurements (benzene as the solvent). The resulting fractional data were used to construct molecular-weight distribution profiles and to calculate the number-average molecular weight (Mn), weight-average molecular weight (Mw), z-average molecular weight (Mz), and the polydispersity index (Mw/Mn) [13].

The cumulative molecular weight distribution was determined as the cumulative sum of the mass fractions of the fractions arranged in ascending order of molecular weight. The differential molecular weight distribution was determined as the individual mass fraction of each fraction, representing the relative content of oligomers with a given molecular weight range. The effective number of fractions was determined based on Shannon entropy, which reflects the degree

Table 1

Production conditions and properties of hydrocarbon resin

Resin	Oligomerization method	Production conditions	Fraction C9 content in reaction mixture, %	Average molecular mass
I	emulsion oligomerization (oil-in-water)	initiator: potassium persulfate; emulsifier: E-30 (C15 alkanesulfonates); reaction temperature: 323 K; reaction time: 3 h	20	490
II			25	520
III			33	535
IV			50	560
V	emulsion oligomerization (water-in-oil)	initiator: benzoyl peroxide; emulsifier: polyglycerol polyricinoleate reaction temperature: 303 K; reaction time: 3 h	66	560
VI			75	575
VII			80	570
VIII	suspension oligomerization	initiator: benzoyl peroxide (two dosages); reaction temperature: 353 K; reaction time: 3 h	33	500
IX		initiator: tert-butylpiperidinomethylperoxide; reaction temperature: 333 K; reaction time: 3 h	25	600
X	initiated oligomerization of fraction C9 after suspension oligomerization	initiator: isopropyl benzene hydroperoxide; reaction temperature: 453 K; reaction time: 6 h	100	655
XI	thermal oligomerization of fraction C9 after suspension oligomerization	reaction temperature: 473 K; reaction time: 6 h	100	630
XII	initiated oligomerization of fraction C9	initiator: di-tert-butyl hydroperoxide; reaction temperature: 453 K; reaction time: 6 h	100	670

of uniformity in the distribution of the mass fractions [14].

Results and discussion

Emulsion oligomerization was carried out in oil-in-water and water-in-oil systems using potassium persulfate and benzoyl peroxide as initiators. The content of the C9 fraction in the reaction mixture was varied from 20 to 80 vol.% to study the effect of the dispersed phase–continuous phase ratio on the molecular-weight distribution (Fig. 1 and 2).

The similarity of differential and cumulative molecular weight distribution curves indicates a narrow molecular weight range of the obtained resins. The main mass fraction (60.4–66.8%) corresponds to the high-molecular-weight component, while low-molecular-weight fractions contribute 2.47–3.11%. Such a distribution confirms the formation of uniform oligomer chains typical for controlled emulsion oligomerization.

With increasing C9 fraction content, both number-average molecular-weight (Mn) and weight-average molecular-weight (Mw) gradually rise

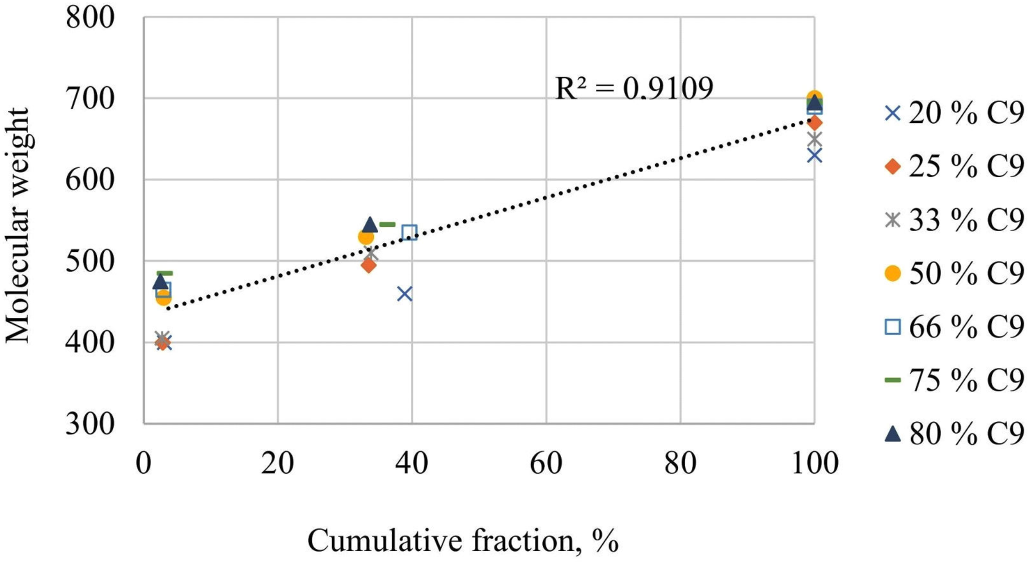

Fig. 1. Cumulative molecular weight distribution curves for hydrocarbon resins **I–VII** as a function of the C9 fraction content in the reaction mixture of emulsion oligomerization

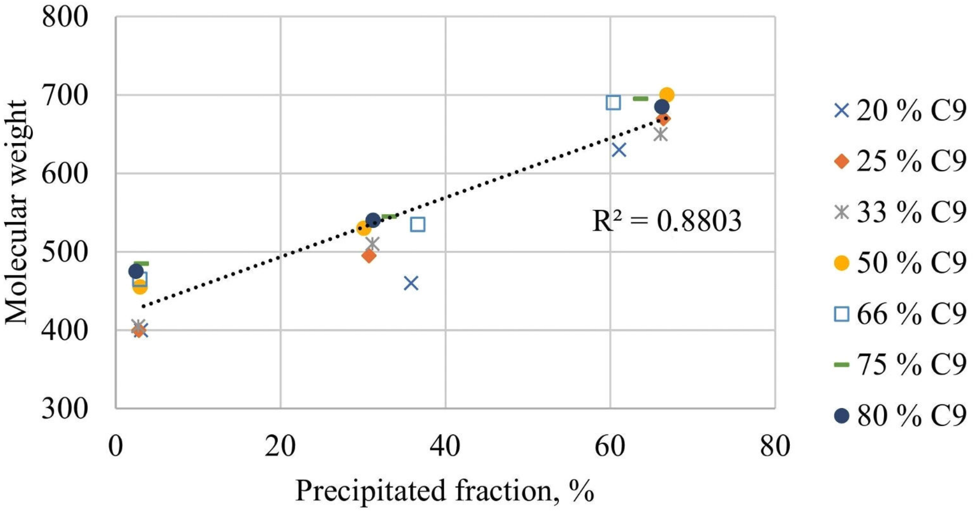

Fig. 2. Differential molecular weight distribution curves for hydrocarbon resins **I–VII** as a function of the C9 fraction content in the reaction mixture of emulsion oligomerization

(Fig. 3). This shows that longer oligomer chains are formed due to a higher monomer concentration in the system. At 50–75% C9 fraction content, the change in Mn and Mw becomes negligible. It indicates a pseudo-stationary regime of oligomer chain growth. Overall, the results confirm that phase composition strongly governs chain-length development in emulsion oligomerization.

Based on the weight-average molecular weight (Mw=575–653) and the molecular weight of styrene, the degree of polymerization of the obtained hydrocarbon resins was estimated to be approximately 5.5–6.3. This indicates that the products mainly consist of oligomeric structures corresponding to hexamers. Polydispersity parameters of hydrocarbon resins **I–VII** obtained by emulsion oligomerization are summarized in Table 2.

The calculated Shannon entropy (0.728–0.776) indicates a moderate level of compositional diversity within the resins.

The normalized entropy (66.2–70.6%) confirms that the mass fractions are distributed relatively uniformly across the oligomer fractions.

The effective number of fractions ranges from 2.07 to 2.17. This means that each resin's composition corresponds to a system with about two balanced fractions.

Entropy analysis (Table 2) shows that variations in the C9 fraction content significantly affect the structural homogeneity of hydrocarbon resins. Higher values of Shannon entropy and the effective number of fractions are observed for hydrocarbon resins obtained at 20% and 66% C9, indicating a more homogeneous fractional composition. Resins synthesized at 25–50% C9 fraction content exhibit slightly lower entropy values, suggesting the predominance of one major oligomeric fraction and a higher degree of structural ordering.

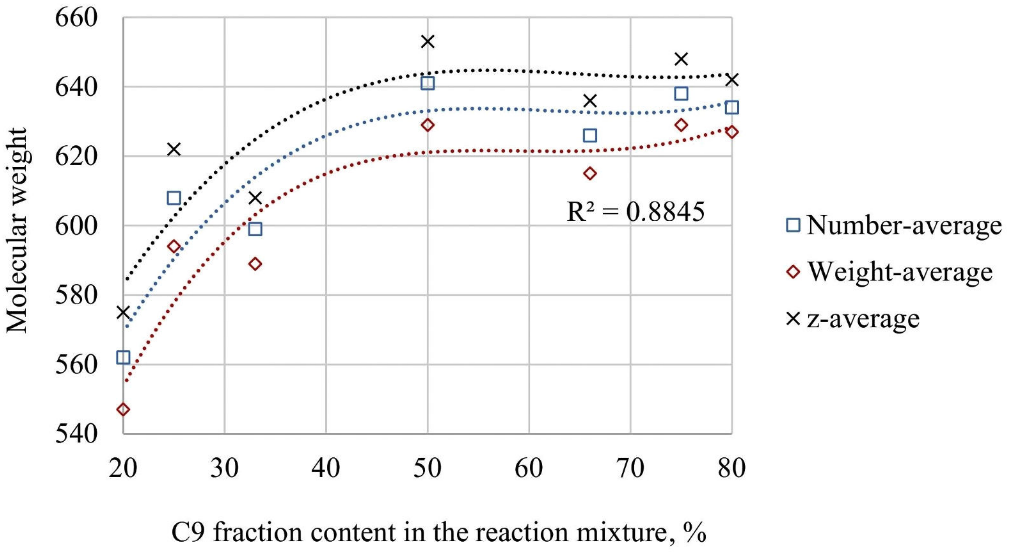

Fig. 3. Molecular-weight averages of resins as a function of C9 fraction content in the reaction mixture

Table 2

Polydispersity parameters of hydrocarbon resins I–VII obtained from C9 fraction by emulsion oligomerization

C9 fraction content in the reaction mixture, %	Shannon entropy	Normalized entropy, %	Effective number of fractions	Polydispersity index (Mw/Mn)
20	0.776	70.6	2.17	1.02
25	0.734	66.9	2.08	1.02
33	0.735	66.9	2.09	1.01
50	0.735	66.9	2.09	1.02
66	0.776	70.6	2.17	1.02
75	0.761	69.3	2.14	1.01
80	0.728	66.2	2.07	1.01

Resin **IX** (suspension oligomerization) shows the steepest and almost linear rise of the curve, indicating minimal heterogeneity and the narrowest molecular-weight range. Resin **VIII** and the cyclopentadiene-based resin **XI** exhibit profiles of intermediate width (Fig. 4). Resin **X**, also obtained via cyclopentadiene oligomerization, displays a sharp increase in the high-molecular-weight region, reflecting a substantial contribution from heavier fractions. The broadest cumulative distribution is observed for the styrene–cyclopentadiene resin XII (formed under high-temperature radical conditions), which shows the most extended high-molecular-weight tail and the highest Mz value, corresponding to the pronounced accumulation of very high molecular-weight species.

The differential curves demonstrate that the resins differ substantially in the width and shape of their molecular-weight distributions (Fig. 5). Resin **IX** (suspension oligomerization) exhibits the narrowest profile: the distribution is concentrated in the region of medium molecular weights with only a minor contribution from low- and high-molecular-weight fractions, indicating a highly uniform product. Resin **VIII** shows a three-component distribution with a noticeable contribution of both medium and high molecular weights, which corresponds to a moderate polydispersity. The cyclopentadiene-based resins (**X** and **XI**) are characterized by a distribution shifted toward higher molecular weights, reflecting the formation of structures with an increased degree of polymerization. The broadest distribution and the most pronounced high-molecular-weight tail are observed for the styrene–cyclopentadiene resin **XII**, which is

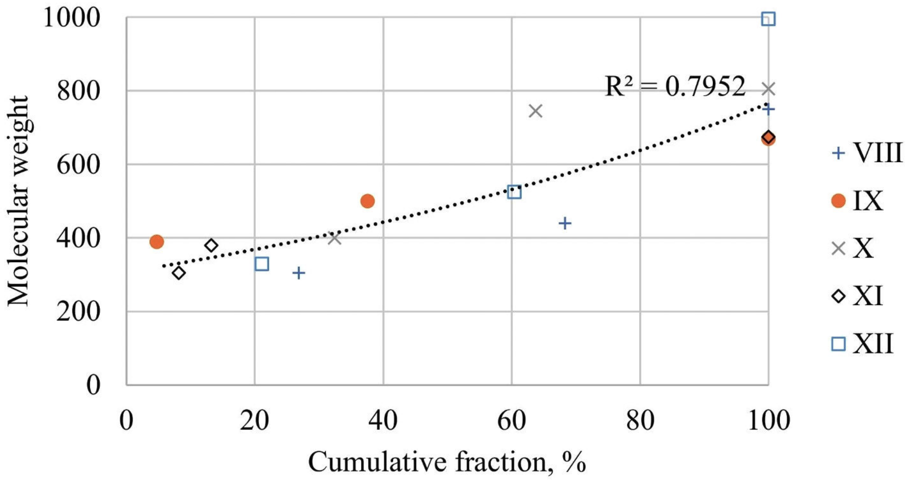

Fig. 4. Cumulative molecular weight distribution curves for hydrocarbon resins **VII–XII**

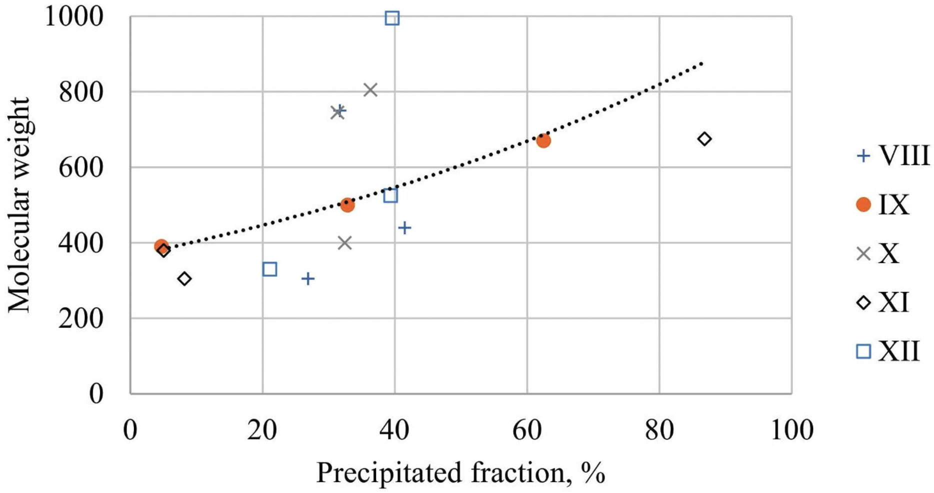

Fig. 5. Differential molecular weight distribution curves for hydrocarbon resins **VIII–XII**

typical for radical processes occurring at elevated temperatures.

The obtained molecular-weight distribution profiles agree with the trends reported for equilibrium telomerization models [11]. These models also show a shift from narrow to broad distributions when chain-growth conditions intensify and the share of high-molecular-weight fractions increases. Polydispersity parameters of styrene resins (**VIII** and **IX**), cyclopentadiene resins (**X** and **XI**), and styrene-cyclopentadiene resin **XII** are presented in Table 3.

The molecular-weight distribution metrics indicate significant differences in resin heterogeneity. Resins **IX** and **XI** exhibit the highest uniformity, as reflected by their low Shannon entropy (0.804 and 0.478) and small effective numbers of fractions (2.24 and 1.62), consistent with narrow distributions (Mw/Mn=1.03–1.06). In contrast, resins **VII**, **X**, and **XII** show substantially broader profiles, with high entropy values (1.06–1.10), normalized entropy near 97–100%, and larger effective number of fractions (≈2.9–3.0). Resin XII displays the greatest heterogeneity (polydispersity index=1.20), confirming the presence of extended high-molecular-weight contributions associated with high-temperature radical oligomerization.

The histogram of molecular-weight averages (Fig. 6) highlights clear differences among the resins. Resins III, V, and IX exhibit closely aligned Mn–Mw values (≈580–630), indicating moderately homogeneous chain growth under low-temperature emulsion or suspension oligomerization conditions.

In contrast, the cyclopentadiene-based resins **X** and **XI**, and especially the styrene–cyclopentadiene resin **XII**, show markedly elevated Mz values (651–781), reflecting the presence of extended high-molecular-weight fractions. Resin **XII** displays the largest separation between Mn, Mw, and Mz, indicating the broadest distribution and the strongest contribution of heavy components.

Overall, low-temperature emulsion and suspension processes produce resins with compact molecular-weight distributions, whereas high-temperature oligomerization in hydrocarbon media generates broader distributions with a pronounced high-molecular-weight contribution.

Table 3

Polydispersity parameters of hydrocarbon resins VIII–XII

Resin	Shannon entropy	Normalized entropy, %	Effective number of fractions	Polydispersity index Mw/Mn
VIII	1.082	98.5	2.96	1.13
IX	0.804	73.1	2.24	1.03
X	1.097	99.8	3.01	1.10
XI	0.478	43.5	1.62	1.06
XII	1.062	96.7	2.91	1.20

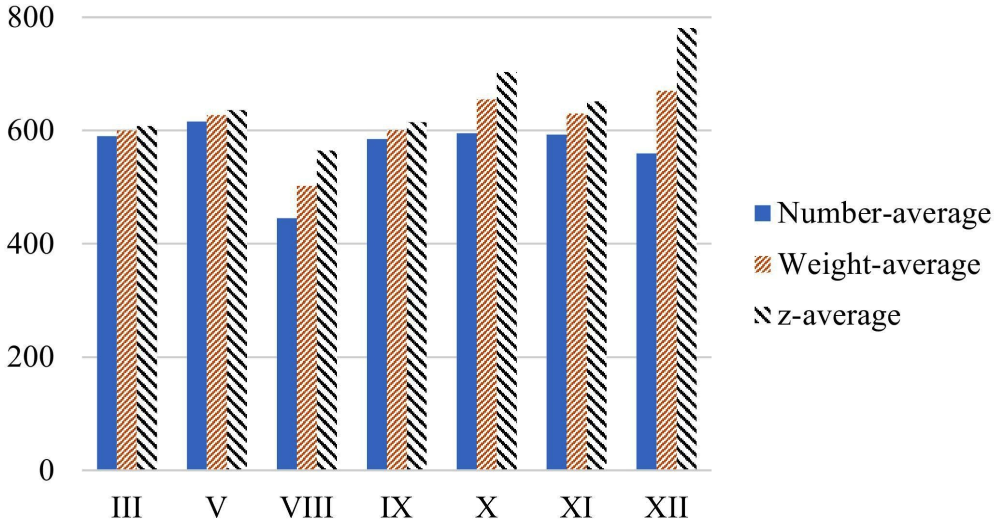

Fig. 6. Molecular-weight averages histogram

Conclusions

The molecular-weight profile of C9 hydrocarbon resins is largely determined by both the oligomerization method and the applied temperature regime. Low-temperature emulsion and suspension processes produce resins with narrow distributions and low polydispersity. High-temperature cyclopentadiene and styrene-cyclopentadiene resins show broad distributions and a high content of heavy fractions. Increasing the proportion of the dispersed phase in emulsions leads to higher average molecular weights. These results show that temperature and phase composition directly control the structural characteristics of C9 resins.

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МОЛЕКУЛЯРНО-МАСОВИЙ РОЗПОДІЛ ВУГЛЕВОДНЕВИХ СМОЛ С9

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У роботі досліджено молекулярно-масовий розподіл вуглеводневих смол, одержаних шляхом олігомеризації фракції С9, рідких побічних продуктів піролізу дизельного палива. Смоли, синтезовані методом емульсійної олігомеризації типу «олія-у-воді» та «вода-в-олії», суспензійної олігомеризації та високотемпературної олігомеризації, фракціонували методом часткового осадження з використанням бензол-етанольних сумішей. Молекулярні маси зразків визначали кріоскопічним методом, а також розраховували середньочислові, середньомасові та z-середні молекулярні маси, показник полідисперсності та ентропійні параметри розподілу. Низькотемпературні емульсійні та суспензійні процеси забезпечили вузькі розподіли з низькою полідисперсністю (1,01–1,13), помірною ентропією (0,80–1,08) та ефективним числом фракцій 2,2–3,0. Збільшення частки дисперсної фази С9 в емульсіях спричиняло зростання середніх молекулярних мас, що свідчить про інтенсифікований ріст ланцюга у більших органічних краплях. Циклопентадієнові смоли, сформовані за високотемпературних умов, характеризувалися підвищеними значеннями молекулярних мас (630–655) та ширшими профілями. Стирен-циклопентадієнова смола, отримана за 453 K, продемонструвала найширший розподіл і найбільш виражений «важкий» високомолекулярний хвіст. Результати показують, що метод олігомеризації та температурний режим визначально впливають на структурну однорідність смол С9: низькотемпературні дисперсійні методи формують більш однорідні матеріали, тоді як високотемпературні радикальні процеси генерують широкі, асиметричні розподіли з домінуванням важких фракцій.

Ключові слова: фракціонування, часткове осадження, молекулярно-масовий розподіл, смола С9, олігомер.

MOLECULAR-WEIGHT DISTRIBUTION OF C9 HYDROCARBON RESINS

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