CHEMMOTOLOGY AND CHEMICAL TECHNOLOGY

UDC 678:67.08

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REGULARITIES OF COOLIGOMERIZATION OF C9 FRACTION AND HEAVY PYROLYSIS TAR MONOMERS

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Abstract. *Regularities of cooligomerization of C9 fraction of diesel fuel pyrolysis and heavy pyrolysis tar with cationic polymerization catalyst - AlCl3, have been investigated. The obtained cooligomer yield and physicochemical properties dependences on the C9 fraction/heavy pyrolysis tar ratio and process conditions have been studied. The optimal conditions have been determined.*

Keywords: C9 fraction; cationic cooligomerization; cooligomers; heavy pyrolysis tar; hydrocarbon resins; liquid pyrolysis products.

1. Problem statement

The lower olefins (ethylene and propylene) production by pyrolysis of hydrocarbon raw materials is related to the problem of by-products utilization. Solving of this problem is an important technical-economic task. The liquid pyrolysis products (LPP) are main by-products, which are formed during gasoline and diesel fuel pyrolysis.

LPP is a mixture of various hydrocarbons, including aromatic hydrocarbons: benzene, naphthalene, acenaphthene, fluorene, phenanthrene, anthracene, their methyl derivatives and other condensed aromatic hydrocarbons; acyclic and alicyclic dienes: isoprene, cyclopentadiene, piperylene etc.; vinylaromatic substances: styrene, methylstyrene etc.; indene and its alkylderivatives, other olefins, as well as admixtures of paraffinic and naphthenic hydrocarbons. These liquid products are important raw material for organic synthesis [4].

LPP are divided into pyrolysis condensate – hydrocarbons mixture boiling off in temperature range 303-473К, and heavy pyrolysis tar (HPT) boiling off at temperatures above 473К [4]. Pyrolysis condensate most often is divided further into separate fractions with narrower boiling ranges: C5 fraction (303-343К), C6-C8 fraction (343-423К) and C9 fraction (423-473К).

Most of HPT hydrocarbons boil off at the temperatures above 473 K. However, due to the indistinctness of condensation HPT also contains some amount of hydrocarbons having final boiling point below 473 K. The composition of the HPT fraction, boiling off at the temperatures below 473 K, is similar to the pyrolysis condensate composition [4]. The HPT yield mainly depends on the feedstock fractional composition and the pyrolysis conditions [4, 6].

C5 and C9 fractions contain considerable amount of polymerizable hydrocarbons and consequently are used to produce cooligomers of aliphatic and aromatic nature, respectively. These cooligomers are called hydrocarbon resins (HR).

2. State of the art

The HR production based on reactive unsaturated hydrocarbons of pyrolysis condensate is commercially realized on industrial scale. It is because of the feedstock affordability, low operating costs, wide use of hydrocarbon resins in paint and varnish, paper making, tire and rubber industries, where they successfully takes the place of plant oils, rosin, albumin and other products of natural origin.

C6-C8 fraction contains very few polymerizable hydrocarbos and are used to obtain pure aromatic substances (benzene, toluene and xylenes) [6].

Bicyclic and tricyclic aromatic hydrocarbons, as well as hydrocarbons with more cycles are dominant components of the HPT. Their content ranges from 51 to 67 % by wt. (table 1).

High content of aromatic hydrocarbons and rather high iodine number (53-56 g I2/100 g) indicates considerable content of unsaturated hydrocarbons and testifies to the HPT liability to polymerization reactions [5].

Though C5 and C9 fractions are conventional raw materials for HR synthesis, HPT can also be used for this aim. HPT is the cheapest fraction of liquid pyrolysis products. That is why use of HPT in the composition of the feedstock for HR synthesis would reduce the resin cost. Use of HPT would also expend HR assortment. Hence, the number of experiments, where the C9 fraction and HPT mixtures of various ratio was used as a raw material, have been carried out. The research was aimed on the optimal C9 fraction/HPT weight ratio determination, as well as on the ascertainment of optimal conditions of HR synthesis from the C9 fraction/HPT mixture of the optimal ratio.

Table 1

HPT composition

Substance name	% by wt.
Naphthalene	14.37
Diphenyl	9.02
2-Methtlnaphthalene	2.45
1- Methtlnaphthalene	2.02
Indene	**1.13**
1-Methyl-2-cyclopropene-1-yl-benzene	**0.94**
2-Methylindene	**0.87**
Methyldicyclopentadiene	**0.82**
Toluene	0.59
3,4,7,7a-tetrahydro-4,7-methano-1H-indene	**0.30**
4-Methyl-1,1'-diphenyl	0.29
Fluorene	0.28
Styrene	**0.23**
1,3-dimethyl-1H-indene	**0.23**
2-ethylnaphthalene	0.21
1,3-cycloheptadiene	**0.20**
1,6-dimethylnaphthalene	0.19
Acenaphthene	0.15
Dimethyldicyclopentadiene	**0.14**
Phenanthrene and anthracene	0.14
1,3-Bis(methylene)-cyclopentane	0.13
1-Methyl-1,2-propadienyl-benzene	**0.13**
3-Methyl-(Z)-1,3,5-hexatriene	**0.11**
1,4-Dimethylnaphtalene	0.11
1-Ethylnaphtalene	0.10
2,3-Dimethylnaphtalene	0.10
9-Methyl-9H-fluorene	0.10
2,7-Dimethylnaphtalene	0.09
Diphenyl-methane	0.09
1-Ethenyl-3-methylene-cyclopentene	**0.06**
3-Methyl-1,1'-biphenyl	0.04
1,4-Dihydronaphtalene	0.61
Non-volatile fraction	~52
Unidentified substances	~10

Nowadays, the following ways of HPT utilization are commercially realized in industrial scale:

– using as a raw material for carbon black production;

– using as a raw material for coking process;

– naphthalene production;

– cycloalkylation of the fraction 473-518 K to obtain synthetic greases;

– cycloalkylnaphthalenes hydration to obtain jet fuel components based on the fraction 473-518 K;

– corrosion-resistant coating production based on high-boiling residual stock;

– dark hydrocarbon resins production [3].

3. Aim of the work

Various substances with electron-seeking properties (Lewis acids) are used as cationic polymerization catalysts. Anhydrous AlCl3 and catalytic complexes based on it are most widely used for hydrocarbon resins synthesis. Anhydrous aluminium chloride has the following advantages over another catalysts of liquid pyrolysis products polymerization: it is affordable, provides high product yield and high polymerization rate, enables using of pyrolysis products with broad boiling range as a raw material and the resins obtained with this catalyst has high softening point [2]. That is why we used this catalyst in our research.

4. Experimental

4.1. Research object

Our research objects include: HPT, composition of which was determined in chromatographic study [7] and is given in table 1, and C9 fraction of diesel fuel pyrolysis liquid products. Polymerizable components content in the C9 fraction is about 60 % by wt. Main of them are styrene (≈ 19 wt %), dicyclopentadiene (≈ 18 wt %) and vinyltoluenes (≈ 8 wt %). Xylenes are main unpolymerizable component of the fraction [1, 4].

Anhydrous AlCl3 was used as a catalyst.

4.2. HR synthesis

Cooligomerization of HPT and C9 fraction was carried out in the three-necked flask fitted with stirrer and back-flow condenser. The flask was placed into the thermostat heated to the process temperature. Cooligomerization was carried out in the temperature range 353–393 K for 0,5–3,5 h under constant intensive stirring. The catalyst content in the reaction mixture varied in the range 0 – 3 % by wt. After the cooligomerization cooligomerizate was sequentially undergone to atmospheric and vacuum distillation to obtain final cooligomer product in residue. Pure C9 fraction, HPT, as well as their mixtures with HPT content varying from 10 to 70% by wt., were used as a raw material.

4.3. Cooligomer properties determination

Bromine number (BN) was determined by iodometric back titration according to common technique. Softening point (SP) was determined by ring and ball method. Average molecular weight of cooligomer was determined by cryoscopic method in benzene. Besides, density of cooligomerizate was determined using areometer.

5. Results and Discussion

5.1. Determination of optimal C9 fraction / HPT ratio

The first stage of the research was aimed on the ascertainment of HR yield and properties dependences on C9 fraction/HPT ratio and choosing of the optimal ratio. The experiments were carried out under the conditions having been determined to be optimal for C9 fraction monomers cooligomerization with anhydrous AlCl3: Ccat. = = 2.5 % by wt, T = 373 K, τ = 3.5 h [1].

As is clear in fig. 1, HR yield is high at any value of C9 fraction/HPT ratio. Anyway, there are two maximums: at 20 % and 70 % by wt. content of HPT.

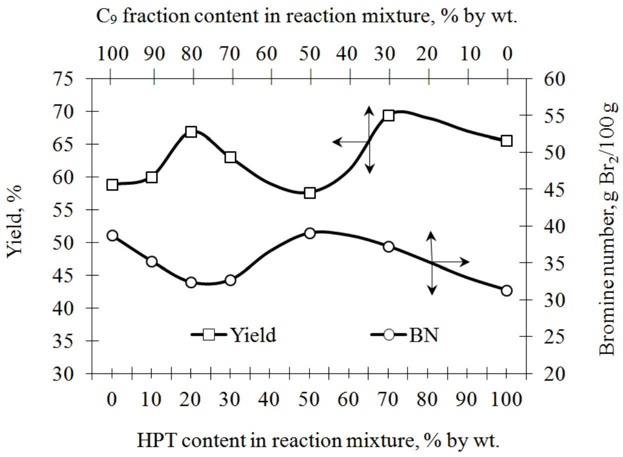

Fig. 1. Dependence of HR yield and bromine number (BN) on the HPT content in the feedstock (the rest is C9 fraction) (AlCl3, Ccat. = 2.5 % by wt., T = 373K, τ = 3.5 h)

To explain the above results (fig. 1) let us consider the approximate composition of C9 fraction and HPT (table 1). The yield of the product obtained from pure C9 fraction indicates complete conversion of polymerizable hydrocarbons. Since HPT contains very few polymerizable compounds – only 5-6 % by wt. (table 1, bold font), and C9 fraction is main source of HR formation, decrease of C9 fraction content in the reaction mixture should result in yield decrease. However, when raising the HPT content from 0 to 20 % by wt. the yield increases. Though HPT contains few polymerizable hydrocarbons, it contains 10-20 % by wt. resinous components [4] and 10-15 % by wt. heavy condensed aromatic compounds – asphaltenes [4], entering the HR composition as they are, without participation in polymerization process. The yield increase is obviously caused by this factor.

The increase of HPT content above 20 % by wt. leads to the resin yield decrease, because negative effect of C9 fraction content reduction surpasses positive effect of presence of asphaltenes and resinous components of HPT.

Besides cooligomerization, alkylation of aromatic, first of all naphthalenic (to a much lesser extent anthracenic and phenanthrenic) hydrocarbons by vinyl and indene monomers is also possible in the system, because, as is known, AlCl3 is an effective alkylation catalyst.

So, the yield increase when raising HPT content over 50 % by wt. is, most probably, caused by alkylation processes intensification. Less value of HR yield when using pure HPT as raw material may be explained by the lack of substances capable of reacting as an alkylating agent (content of such substances in HPT does not exceed 3 % by wt. (table 1, numbers in bold).

Bromine number of the HR is high in whole studied range of the feedstock compositions, and varies in relatively narrow range: 31.3–39.1 g Br2/100 g. The most unsaturated product is obtained from pure C9 fraction and from the mixture where C9 fraction/HPT ratio is 50/50 (fig. 1). The cooligomer obtained from C9 fraction is highly unsaturated due to the presence of dicyclopentadiene (DCPD) units involving double bonds. Reduce of C9 fraction content in the feedstock leads to decrease of the DCPD fragments content in HR resulting in HR bromine number decrease. However, unsaturation starts rising again when HPT content is raised above 30 % by wt. This is probably related to the unsaturation of the resinous compounds present in HPT composition, which affect the cooligomer bromine number when raising HPT content in the feedstock. At HPT content above 70 % by wt., the reactions of aromatic compounds alkylation with unsaturated substances prevail over other processes, and HR of lower unsaturation are consequently formed.

Average molecular weight and softening point of cooligomer decrease (fig. 2) when reducing the C9 fraction/HPT ratio. Average molecular weight of the HR obtained from HPT is 487 only, while the HR obtained under the same conditions from C9 fraction is characterized by molecular weight of 1094.

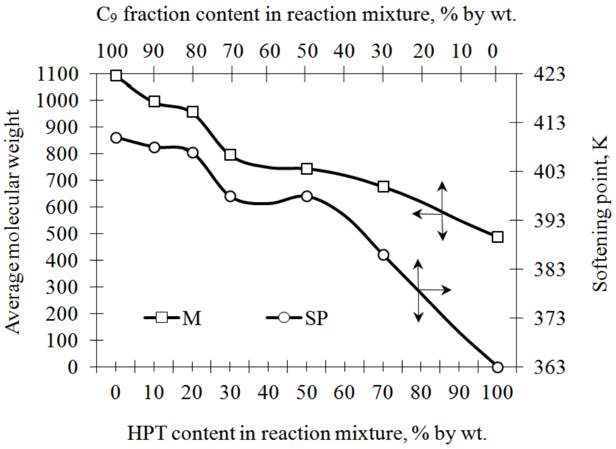

Fig. 2. Dependence of HR average molecular weight (M) and softening point (SP) on the HPT content in the feedstock (the rest is C9 fraction) (AlC13, Ccat. = = 2.5 % by wt, T = 373K, τ = 3.5 h)

Such regularities conform well to the conclusions made above when analyzing HR yield dependence on the feedstock composition. If HR is mostly a product of C9 fraction polymerizable components polymerization, than it, obviously, has higher molecular weight compared to that of a resin consisting mostly of naphtalenic hydrocarbons alkylation products and resinous compounds present in the initial HPT.

Sharp decrease of HR softening point (sharper than corresponding decrease of molecular weight), when raising HPT content above 50% by wt., indicates the cooligomer nature change. At the HPT content below 50 % by wt., the resulting HR is mostly styrene-DCPD cooligomer with admixtures of asphaltenes and resinous components of HPT, while at the HPT content above 50 % by wt., the resulting HR mostly consists of the products of aromatic compounds alkylation with styrenic and indenic monomers, asphaltenes and resinous components of HPT.

The highest HR yield is achieved at the C9 fraction/HPT ratio equal to 30/70 (69.4 %) and somewhat less (66.9 %) at 80/20. However, HR molecular weight is considerably higher at the C9 fraction/HPT ratio = 80/20 (957 compared to 677 at 30/70). Consequently, softening point is higher as well (407 K compared to 386 K at 30/70). HR bromine number varies in relatively narrow range (31.3–39.1 g Br2/100 g), so it cannot be an optimality criterion. Based on the foregoing discussion the C9 fraction/HPT ratio equal to 80/20 is considered to be the optimal ratio.

Further research is aimed on the ascertainment of optimal conditions of HR synthesis from the C9 fraction and HPT mixture at the optimal ratio.

5.2. Determination of optimal conditions of HR synthesis from the mixture: 80% by wt C9 fraction + 20 % by wt. HPT

5.2.1. HR yield and properties dependence on the process temperature

In case of HR synthesis from C9 fraction with anhydrous AlC13 as a catalyst the catalyst concentration – 2.5 % by wt. and cooligomerization duration – 3.5 h have been determined to be optimal conditions [1]. That is why these conditions were applied when studying temperature influence on the process of HR synthesis from the C9 fraction/HPT (80/20) mixture.

As is clear in fig. 3, maximum HR yield is achieved at 373 K. Further raise of the process temperature results in the cooligomer yield decrease.

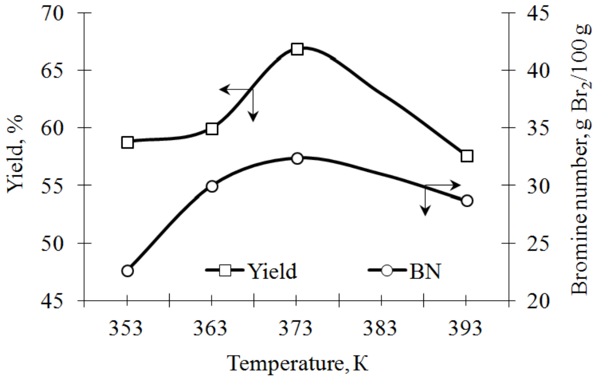

Fig. 3. Dependence of HR (obtained from C9 fraction/HPT mixture (80/20)) yield and bromine number (BN) on the process temperature (Ccat. = = 2.5 % by wt, τ = 3.5 h)

The yield decrease at temperatures over 373 K, is, probably, caused by intensification of chain termination reactions, resulting in formation of considerable amount of low-molecular cooligomers (dimers, trimers). These low-molecular cooligomers are partially distilled off the cooligomerizate at vacuum stripping, which is carried out under rather severe conditions (T = 453K, p = 15-20 mm Hg). In addition, isomerization and alkylation reactions probably take place at high temperatures. These reactions almost do not contribute to the yield increase and just cause unproductive use of some part of the catalyst.

The temperature dependence of the cooligomer unsaturation is also characterized by a maximum (fig. 3). The process temperature raise from 353 to 373K causes bromine number increase by 10 g Br2/100 g, while temperature raise by another 20K results in HR unsaturation decrease.

The HR of highest average molecular weight and softening point is obtained at 373K (fig. 4).

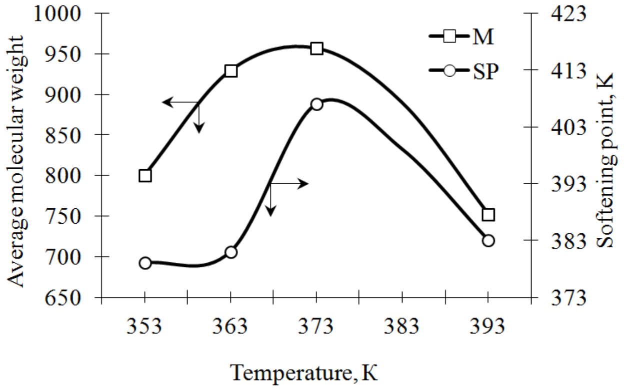

Fig. 4. Dependence of HR (obtained from C9 fraction/HPT mixture (80/20)) average molecular weight (M) and softening point (SP) on the process temperature (Ccat. = 2.5 % by wt., τ = 3.5 h.)

The decrease of these characteristics at temperatures above 373 K indicates rapid termination of the propagating cooligomer chain and testifies to the supposition made to explain corresponding yield decrease (fig. 3). It worth to be noted that temperature raise from 353 to 363 K results in substantial increase of molecular weight but has almost no effect on the softening point, while temperature raise form 363 to 373 K, on the contrary, causes sharp increase of softening point whereas average molecular weight increase is insignificant. This indicates certain changes in HR structure and composition take place when the temperature increases.

Thus, it may be concluded that the temperature of 373 K makes it possible to obtain the product not only with maximum yield but also of maximum molecular weight, softening point and unsaturation that is desirable for HR. So, the optimum cooligomerization temperature is 373 K. In consideration of this, further research was carried out at this temperature.

5.2.2. HR yield and properties dependence on the catalyst concentration

The catalyst concentration increase results in the product yield increase, which reaches the maximum at 2.5 % by wt. of the catalyst in the reaction mixture (fig. 5). Further raise of the catalyst concentration proves no positive effect and, thus, is not reasonable. The HR yield increase is most sharp when raising the catalyst concentration up to 1.5 % by wt, whereupon it decelerates.

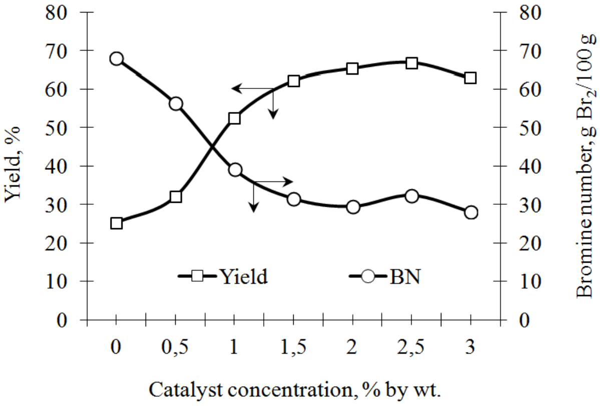

Fig. 5. Dependence of HR (obtained from C_9 fraction/HPT mixture (80/20)) yield and bromine number (BN) on the catalyst concentration (T = 373 K, τ = 3.5 h)

HR bromine number decreases when raising the catalyst concentration (fig. 5). However, the similar way as for the yield, substantial effect is observed up to 1.5% by wt. only, and further increase of the catalyst concentration has no considerable impact on the HR unsaturation.

Raise of the catalyst concentration results in the HR average molecular weight and softening point increase (fig. 6). Herewith three regions may be distinguished:

1) from 0 to 0,5 % by wt – dramatic increase of molecular weight and softening point;

2) from 0,5 to 1,5 % by wt – plateau – region wherein the catalyst concentration increase almost does not affect the molecular weight and softening point values;

3) from 1,5 to 2,5 % by wt – dramatic increase of molecular weight and softening point when raising the catalyst concentration.

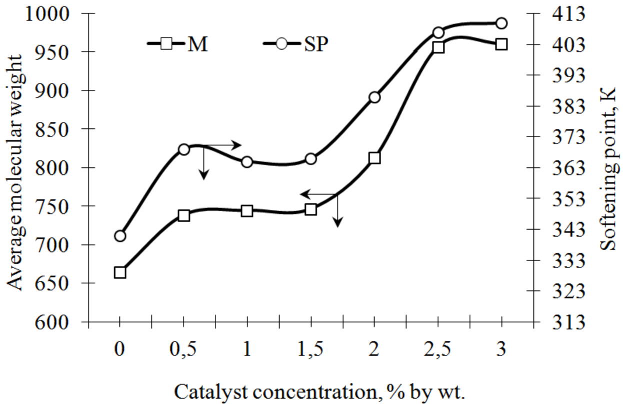

Fig. 6. Dependence of HR (obtained from C_9 fraction/HPT mixture (80/20)) average molecular weight (M) and softening point (SP) on the catalyst concentration ($T = 373$ K, $\tau = 3.5$ h)

The first region represents transition between two HR synthesized in fact by different methods: thermal cooligomerization (occurring by radical mechanism) and catalytic cooligomerization (occurring by cationic mechanism). As is clear from fig. 6, the catalytic method allows achieving higher molecular weight even at insignificant concentration of the catalyst.

In the plateau region molecular weight increase being observed in region (1) is levelled off due to the fact that more cooligomer chains are simultaneously initiated when raising the catalyst concentration and this leads to molecular weight decrease.

DCPD present in the raw material involves two double bonds enabling branched and cross-linked structures formation, which are characterized by high molecular weight. But the cooligomerization by both double bonds becomes possible only when great amount of catalyst is used. That is why it may be supposed that at the catalyst concentration up to 1.5% by wt. linear cooligomers are mostly formed, while at higher concentration monomers addition by double bonds of DCPD units is also occurs, resulting in branched structures formation. However, due to the limited number of DCPD units such processes occur to a certain extent and molecular weight and softening point increase is no more observed when raising the catalyst concentration over 2.5 % by wt.

Therefore, the optimum concentration of $AlCl_3$ in the reaction mixture is 2.5 % by wt., because the maximum yield is achieved at this concentration, and the resulting HR is characterized by the highest average molecular weight and softening point. However, if high molecular weight is not a crucial requirement, the catalyst concentration of 1-1.5 % by wt. may be considered to be optimal, because the concentration raising above this value results in very slight increases of the product yield. In addition using of less amount of the catalyst is desirable as it reduces HR cost.

5.2.3. HR yield and properties dependence on the process duration

The research of the cooligomerization duration effect was carried out at the optimum temperature (373K) and two concentrations of the catalyst: 1 and 2.5 % by wt.

Though the HR yield increases when raising the duration, this increase is very slight and we can state that the cooligomerization duration is the factor least affecting the product yield (compared to temperature and, especially, the catalyst concentration). It can also be stated that the cooligomerization with $AlCl_3$ occurs very rapidly, and the higher the catalyst concentration the more rapid the process is, and the less dependent on the duration the yield is (fig. 7). Thus, at the catalyst concentration 2.5 % by wt. the product yield of 59.9% is reached in 0.5 h., and duration raise by another 3 h. (7 times) results in the yield increase by 5% only. At the catalyst concentration 1% by wt. the cooligomer formation occurs not so rapidly and the process duration raise affects the product yield much stronger: increase of cooligomerization duration from 0.5 to 3.5 h. leads to HR yield increase from 32.2 to 52.6%.

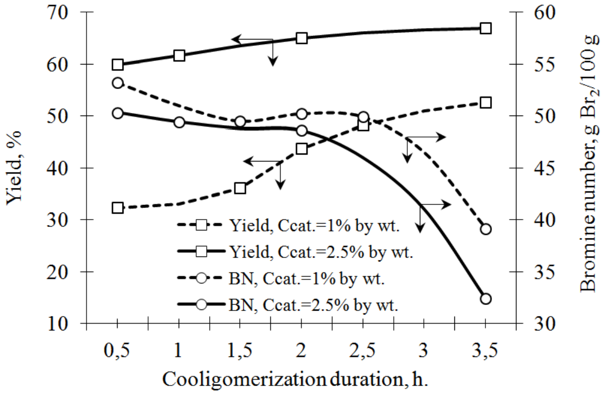

Fig. 7. Dependence of HR (obtained from C_9 fraction/HPT mixture (80/20)) yield and bromine number (BN) on the cooligomerization duration ($C_{cat.} = 2.5$ % by wt, $T = 373$K)

HR bromine number decreases when raising cooligomerization duration. And this decrease is most dramatic when the duration exceeds 2.5 h. (fig. 7), and it is sharper in case of higher concentration of the catalyst (2.5 % by wt). This fact agrees with the supposition made to explain molecular weight increase when raising the catalyst concentration over 2 % by wt.

When raising the cooligomerization duration molecular weight of the HR decreases at first (from 0.5 to 2 h), and then (from 2 to 3.5 h) remains almost constant. Such regularity is observed in case of both 1 % by wt. and 2.5 % by wt. concentration of the catalyst (fig. 8).

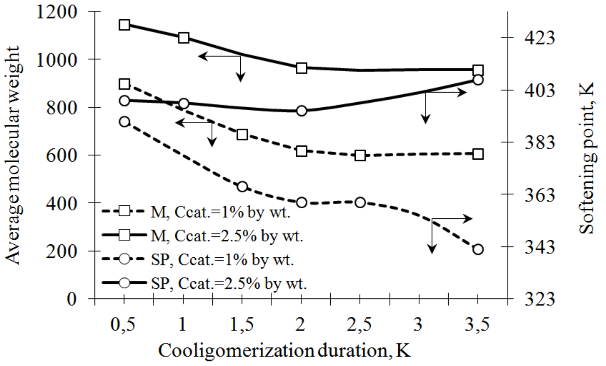

Fig. 8. Dependence of HR (obtained from C_9 fraction/HPT mixture (80/20)) average molecular weight (M) and softening point (SP) on the cooligomerization duration ($C_{cat.} = 2.5$ % by wt., T = 373K)

Seemingly, molecular weight should increase in time, as propagating cooligomer chains can reach larger size in longer time. However, the influence of this factor is negligible, because most of the HR is formed in the first 0.5-1 h. of the cooligomerization (fig. 7).

Thus, the optimal conditions of the HR synthesis from the mixture of 80% by wt. C_9 fraction and 20% by wt. HPT with anhydrous $AlCl_3$ as a catalyst are as follows:

– the catalyst concentration – 2.5 % by wt;
– the process temperature – 373K;
– the cooligomerization duration – 2 h.

6. Conclusions

Based on the research done the regularities of cooligomerization of C_9 fraction and HPT of liquid pyrolysis products catalyzed by $AlCl_3$ have been ascertained. It has been determined how the product yield and properties depend on the C_9 fraction/HPT ratio. The optimum C_9 fraction/HPT ratio in terms of HR yield and properties has been determined to be equal 80/20. Study of HR synthesis from such mixture under various conditions allowed as to choose the optimum cooligomerization conditions, which are as follows: the catalyst concentration – 2.5 % by wt, duration – 2 h., temperature – 373 K.

The HR obtained at these conditions are characterized by the yield – 65.0 %, average molecular weight – 967, softening point – 395 K and bromine number – 48.6 g Br_2/100 g.

The high value of the HR softening point makes it possible to use such HR in protective and corrosion-resistant coatings, for bitumen modification etc.

High unsaturation of the HR enables their further modification to obtain functionalized hydrocarbon resins with broad application area.

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Received 10 July 2014.

З. Я. Гнатів[1], І. Є. Никулишин[2], Т. О. Ворончак[3], Г. М. Рипка[4]. Закономірності коолігомеризації мономерів фракції C_9 та важкої смоли піролізу

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Досліджено закономірності коолігомеризації фракції C_9 піролізу дизельного палива з важкою смолою піролізу в присутності каталізатора катіонної полімеризації – $AlCl_3$. Вивчено залежність виходу та фізико-хімічних властивостей отриманих коолігомерів від співвідношення фракція C_9 / важка смола піролізу та умов процесу. Встановлено оптимальні умови.

Ключові слова: важка смола піролізу; катіонна коолігомеризація; коолігомери; нафтополімерні смоли; рідкі продукти піролізу; фракція C_9.

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Исследованы закономерности соолигомеризации фракции C_9 пиролиза дизельного топлива с тяжелой смолой пиролиза в присутствии катализатора катионной полимеризации – $AlCl_3$. Изучена зависимость выхода и физико-химических свойств полученных соолигомеров от соотношения фракция C_9/тяжелая смола пиролиза и условий процесса. Установлены оптимальные условия.

Ключевые слова: жидкие продукты пиролиза; катионная соолигомеризация; нефтеполимерные смолы; соолигомеры; тяжелая смола пиролиза; фракция C_9.

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