

The Effect of Modification Method of ZSM-5 Zeolite with Zinc on the Product Composition in Cracking of Hydrocarbon Feedstock

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Abstract—The influence of the zinc incorporation method (ion exchange, impregnation) and its content (from 0.25 to 0.75 wt %) in ZSM-5 zeolite on the physicochemical characteristics of the zeolite (textural parameters, concentration of acid sites) and on the catalytic activity of cracking catalysts with the additives containing the modified zeolites in transformations of hydrotreated vacuum gasoil was studied. The zinc introduction by impregnation leads to a slight decrease in the zeolite specific surface area and micropore volume from 420 to 380 m²/g and from 0.131 to 0.117 cm³/g, respectively, and the total concentration of acid sites at the maximal zinc content increases by 17% compared to unmodified ZSM-5. Modification of the zeolite samples with zinc by ion exchange does not alter the main pore structure parameters compared to the initial zeolite, whereas the acid site concentration reaches the highest values (up to 2025 μmol/g). With an increase in the zinc content of ZSM-5 zeolite, with both modification methods (ion exchange, impregnation), the yield of the gasoline fraction and aromatic hydrocarbons in liquid products of cracking of hydrotreated vacuum gasoil increases.

Keywords: ZSM-5 zeolite, zinc, modification, ion exchange, impregnation, catalytic cracking, aromatic hydrocarbons

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The production of gasoline with high octane number is a topical problem for oil refineries. This goal can be accomplished by varying the content of olefins, isoparaffins, or aromatic compounds in compounding gasolines. The gasoline fraction from catalytic cracking is characterized by relatively high octane number due to the presence of unsaturated hydrocarbons, but their content is restricted because of low stability of the resulting fuels. Another way to increase the gasoline octane number is increasing the fraction of aromatic hydrocarbons and isoparaffins.

Numerous studies have shown that ZSM-5 zeolite with its pore system, acid properties, and high thermal and hydrothermal stability is an excellent component of a cracking catalyst [2, 3]. However, ZSM-5 in the H-form poorly meets the conditions for using it as a component of a cracking catalyst because of several factors. First,

the acidity should be on the level when it already ensures cracking of C–C bonds but does not yet promote secondary reactions: hydrogen transfer leading to low yield of light olefins and coking promoted by strong acid sites. Second, the pore size (about 0.5 nm in diameter) in HZSM-5 is equivalent to the size of reactant molecules, which can give rise to diffusion limitations and cause pore blocking with coke deposits [4]. Several approaches are widely used for improving the catalytic characteristics of ZSM-5. These include control of the zeolite acidity [5, 6], synthesis of nanocrystalline [7, 8] and hierarchic zeolites [4, 9–13], and modification by introducing phosphorus [14–16] and metals (Zn, Fe, Ga, Ca) [17–19]. The use of modified ZSM-5 zeolite as an additive to the cracking catalyst favors an increase not only in the yield of light olefins, but also in the content of aromatic hydrocarbons in the liquid products [17, 20–22].

The effect of the ZSM-5 zeolite modification with zinc was studied in detail for the aromatization of alkanes [23]. According to [24, 25], introduction of zinc into ZSM-5 zeolite allows an increase in the fraction of aromatic hydrocarbons due to suppression of cracking and promotion of dehydrogenation. The presence of Zn compounds leads to substantial changes in the acid properties of ZSM-5, namely, to a decrease in the density of Brønsted acid sites in proportion with the zinc content and to the formation of new relatively strong Lewis acid sites [26]. However, the zinc content of ZSM-5 zeolite should be limited, because high zinc content leads to rapid coking of the catalyst; as a result, its activity decreases. Data on introduction of ZSM-5 zeolite modified with metals into the cracking catalyst formulation are scarce, and this is a topical problem. Such modification of the cracking catalyst can increase the yield of aromatic hydrocarbons, thus increasing the research octane number of gasoline [1]. The procedure for Zn introduction into a zeolite strongly influences the acid properties of the final catalysts and the composition of the products obtained on them [32, 33].

In this work, we studied the effect of ZSM-5 zeolite modification with zinc on the formation of aromatic compounds in cracking product.

EXPERIMENTAL

Preparation of zeolite and catalyst samples.

Samples of ZSM-5 zeolite (Zeolyst International, $\text{SiO}_2/\text{Al}_2\text{O}_3 = 23$) with different zinc content (0.25, 0.5, and 0.75 wt %) were prepared by two methods: ion exchange and impregnation with a zinc nitrate solution. In the first case, the ammonium form of ZSM-5 zeolite was treated with an aqueous $\text{Zn}(\text{NO}_3)_2$ solution for 3 h at 80°C, the suspension was filtered, and the product was dried at 100°C. The H-form of the zeolite was impregnated using a Buchi Mini Spray Dryer B-290 device by addition of the calculated amount of $\text{Zn}(\text{NO}_3)_2$ to a zeolite suspension, followed by spray drying. Then, the zeolites were dried at 100°C and calcined at 550°C.

From the zeolite samples obtained, we prepared additives to a cracking catalyst. The additives consisted of bentonite clay, aluminum oxide, and modified ZSM-5 zeolite. The components were taken in 1 : 1 : 2 ratio. The additives were dried at 100°C and then calcined for 5 h at 600°C. Then, the additives were treated for 5 h at 788°C in 100% steam in accordance with ASTM D 4463. The

catalyst-additive system was prepared by mechanical mixing of the preliminarily stabilized AV cracking catalyst with 10 wt % zeolite-containing additive.

Study of catalyst components. The chemical composition of the samples was determined by atomic absorption spectrometry with a Shimadzu AA-6300 device after the zeolite breakdown in solutions of mineral acids (sulfuric, hydrochloric, and hydrofluoric). The main characteristics of the pore structure of the samples were determined from the isotherms of the nitrogen adsorption-desorption at -196°C , measured with an ASAP-2020 volume vacuum static installation (Micromeritics, the United States). The specific surface area (S_{sp}) was determined by the BET method. The specific micropore volume was estimated by the comparative method. The total amount of acid sites in the zeolites was determined by temperature-programmed desorption (TPD) of ammonia with an AutoChem-2920 high-precision chemisorption analyzer (Micromeritics). The coordination state of aluminum in zeolites was studied by ^{27}Al MAS NMR with an Advance 400 device (Bruker).

Kinetic trials. Hydrotreated vacuum gas oil containing 300–350 ppm sulfur and 650–700 ppm nitrogen was used as a real feedstock. The catalytic properties of the samples were studied using a flow-through laboratory installation with a fixed bed of MAK-10 catalyst in accordance with ASTM D 3907 at 527°C. The gaseous products were analyzed with a GK-1000 gas chromatograph equipped with a capillary column (SiO_2 , 30 m \times 0.32 mm) and a flame ionization detector. The quantitative analysis of liquid cracking products was performed in accordance with ASTM D 2887 (simulated distillation method) with a GC2010 chromatograph (Shimadzu) equipped with an Rtx-2887 capillary column (10 m \times 0.53 mm \times 2.65 μm) and a flame ionization detector. All the liquid products with the boiling point lower than 216°C were assigned to the gasoline fraction. The component composition of the liquid products was determined with a Shimadzu GCMS-QP2010 gas chromatograph-mass spectrometer equipped with an HP-1 ms column (60 m \times 0.25 μm) and an additional flame ionization detector.

The coke content on the catalyst was determined from the weight loss upon calcination of the catalyst sample in air at 550°C.

The contribution of the intermolecular hydrogen transfer was quantitatively estimated using the hydrogen transfer coefficient reflecting the formation selectivity

Table 1. Chemical composition of ZSM-5 samples used for preparing an additive to the cracking catalyst

Zeolite sample	Zn introduction procedure	Content, wt %		
		Na ₂ O	Al ₂ O ₃	ZnO
HZSM-5	–	0.07	6.18	–
1-ZSM-5-ie	Ion exchange	0.05	5.98	0.36
2-ZSM-5-ie		0.06	6.70	0.66
3-ZSM-5-ie		0.03	6.03	0.86
4-ZSM-5-im	Impregnation	0.11	6.98	0.29
5-ZSM-5-im		0.09	7.15	0.61
6-ZSM-5-im		0.07	7.22	0.81

ratio of butanes and butylenes [27]. The hydrogen transfer coefficient (HTC) was calculated by the formula:

$$\text{HTC} = \frac{S_{\text{butanes}}}{S_{\text{butylenes}}},$$

where HTC is the hydrogen transfer coefficient; S_{butanes} , $S_{\text{butylenes}}$, selectivity of the formation of butanes and butylenes, respectively.

RESULTS AND DISCUSSION

Physicochemical Characteristics of Modified Zeolites

Chemical composition. All the zeolite samples are characterized by low sodium oxide content. The zinc oxide content was 0.36, 0.66, and 0.86 wt % for the samples prepared by ion exchange and 0.29, 0.61, and 0.81 wt % for those prepared by impregnation (Table 1).

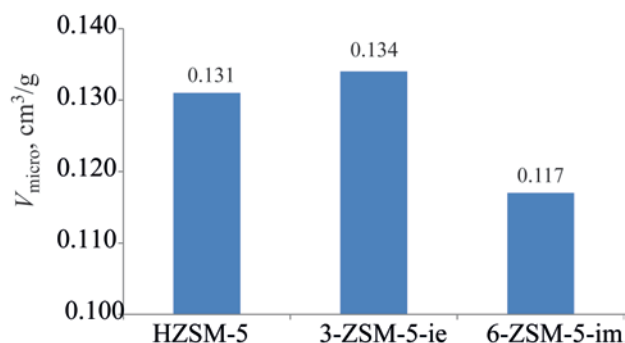


Fig. 1. Influence of the zinc introduction method on the micropore volume of ZSM-5 zeolite.

Textural properties, coordination of aluminum atoms, and concentration of acid sites. The apparent BET specific surface area, total adsorption pore volume, and specific micropore volume of the initial sample (before modification) are 420 m²/g, 0.218 cm³/g, and 0.131 m³/g, respectively.

Introduction of zinc by ion exchange does not lead to textural changes. Introduction of zinc into ZSM-5 zeolite by impregnation leads to a slight decrease (by up to 10%) in the specific surface area (from 420 to 380 m²/g) and in the specific micropore volume, probably due to blocking of micropores with (ZnO)_n clusters [22] (Fig. 1).

The ²⁷Al NMR spectra of the zeolite samples in the NH₄-form (initial) and H-form (calcined at 550°C) and with zinc introduced by ion exchange and impregnation are shown in Fig. 2. All the samples of ZSM-5 zeolites show a peak in the region of 60 ppm, corresponding to the tetrahedral coordination of aluminum atoms. Upon calcination of the initial zeolite at 550°C, a peak corresponding to the octahedral coordination appears in the region of 0 ppm. This fact suggests partial breakdown of the zeolite crystal lattice and formation of nonstructural aluminum whose fraction is about 10–15%. Nonstructural aluminum is also formed in the zeolite sample modified with zinc by impregnation. The zinc introduction by ion exchange does not lead to the formation of aluminum in octahedral coordination; i.e., the zeolite crystal lattice is preserved. The data obtained suggest that the zinc introduction into ZSM-5 zeolite by ion exchange leads to stabilization of the framework, enhancing the thermal stability of the sample (similarly to the action of REE in Y zeolite).

Figure 3 shows the temperature profiles of the ammonia desorption for zeolite samples with the maximal zinc oxide content and for the initial HZSM-5. Two

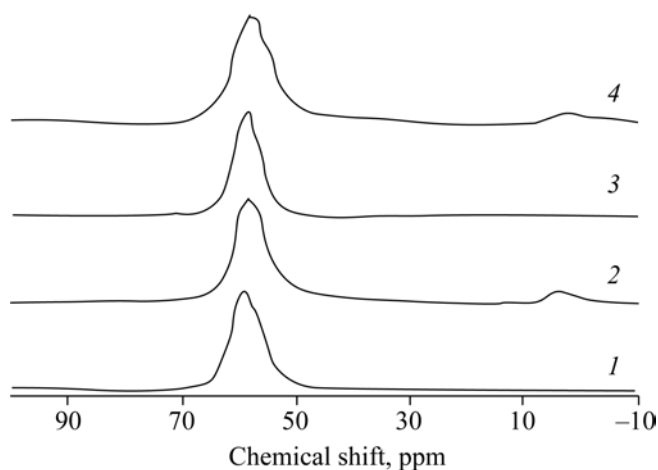


Fig. 2. ^{27}Al NMR spectra of zeolites: (1) $\text{NH}_4\text{ZSM-5}$, (2) HZSM-5, (3) 3-ZSM-5-ie, and (4) 6-ZSM-5-im.

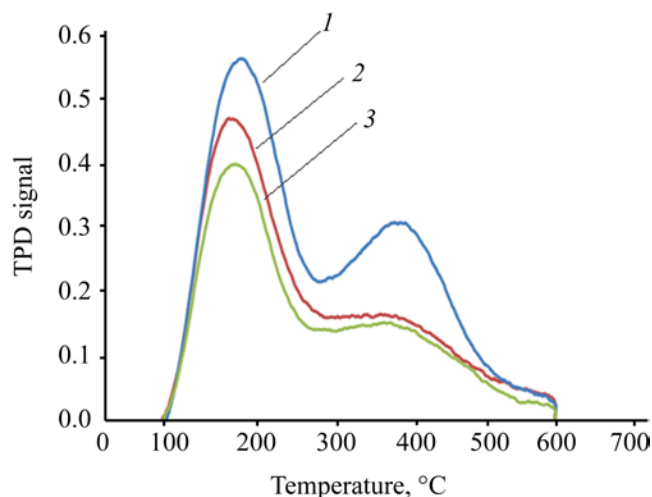


Fig. 3. TPD- NH_3 profiles of zinc-modified ZSM-5 samples: (1) 3-ZSM-5-ie, (2) 6-ZSM-5-im, and (3) HZSM-5.

temperature intervals corresponding to weak and strong acid sites are clearly seen: 100–300 and 300–600°C. Introduction of zinc into ZSM-5 zeolite favors an increase in the amount of both weak (low-temperature interval) and strong (high-temperature interval) acid sites. A gradual increase in the content of zinc oxide introduced into ZSM-5 zeolite by impregnation leads to an increase in the concentration of acid sites from 1230 to 1484 $\mu\text{mol/g}$ without redistribution with respect to the acid site strength. The TPD- NH_3 profile for the 6-ZSM-5-im sample is similar to that for the initial zeolite, with the main maximum (208°C) in the interval of weaker acid sites (100–300°C). In the TPD- NH_3 profile for the 3-ZSM-5-ie sample, along with an increase in the intensity of the low-temperature peak, the high-temperature peak in the interval 300–600°C with a maximum at 430°C becomes pronounced. The modifier introduction into ZSM-5 zeolite by ion exchange ensures the largest increase in the concentration of acid sites (by more than 60%): from 1230 to 2025 $\mu\text{mol/g}$. An increase in the acidity of the zinc-modified zeolite may be due to preservation of the zeolite crystal structure and to the formation of $-\text{Al}-\text{O}-\text{Zn}-\text{O}-\text{H}$ sites acting as Brønsted acid sites (BASs) and of strong Lewis acid sites (LASs) [26]. It should be emphasized that, because of low zinc content of the modified zeolite, the probability of the formation of $\text{O}-\text{Al}-\text{O}-\text{Zn}-\text{O}-\text{Al}$ bonds (leading, apparently, to a decrease in the concentration of acid sites), described in [34, 35], is low.

Influence of the zinc content and introduction method on the product composition in cracking of hydrotreated vacuum gas oil in the presence of the “additive + catalyst” system. According to published data, modification of ZSM-5 zeolite with zinc allows efficient variation of its acidity and accelerates the formation of aromatic compounds on surface Lewis acid sites owing to enhancement of the hydrogen transfer [28–31].

Tables 2 and 3 show the material balance for the cracking of vacuum gas oil on catalysts mixed with additives containing ZSM-5 zeolites modified with zinc by ion exchange and impregnation, respectively.

The introduction of the additive containing zinc-modified ZSM-5 zeolite into the cracking catalyst formulation leads to a decrease in the yield of light olefins from 17.8 to 15.3 wt % when using the zeolite modified by ion exchange and to 16.8 wt % for the zeolite modified with impregnation. In particular, the fraction of propylene also decreases for the samples with the zeolite modified by ion exchange, which is due to an increase in the contribution of intermolecular hydrogen transfer (Table 2).

With an increase in the fraction of zinc oxide introduced by ion exchange and impregnation in ZSM-5 zeolite, the yield of the gasoline fraction in cracking of hydrotreated vacuum gasoil increases, which is due to an

Table 2. Catalytic properties of catalyst–additive (10 wt %) systems containing ZSM-5 zeolites modified with zinc by ion exchange (trials according to ASTM D 3907; 527°C; feedstock: hydrotreated vacuum gas oil; catalyst : feed ratio 4; feeding time 30 s)

Sample	HZSM-5	1-ZSM-5-ie	2-ZSM-5-ie	3-ZSM-5-ie
Zn content of ZSM-5 zeolite, wt %	0	0.36	0.66	0.86
Yield of products, wt %				
Dry gas	1.8	2.0	2.0	2.1
Liquefied hydrocarbon gas, including:	26.1	26.3	25.8	24.4
isobutane	6.6	6.7	6.4	6.9
C ₃ –C ₄ olefins	17.8	18.0	17.8	15.3
propylene	8.4	8.3	8.3	7.5
Gasoline (s.b.–216°C)	45.9	46.5	47.6	48.4
Light gasoil (216–350°C)	15.3	14.5	13.5	13.5
Heavy gasoil (350°C–e.b.)	5.7	5.2	5.9	5.3
Coke	5.2	5.5	5.3	6.3
Conversion, wt %	79.0	80.3	80.7	81.2
Hydrogen transfer coefficient	0.894	0.899	0.861	1.199

Table 3. Catalytic properties of catalyst–additive (10 wt %) systems containing ZSM-5 zeolites modified with zinc by impregnation (trials according to ASTM D 3907; 527°C; feedstock: hydrotreated vacuum gas oil; catalyst : feed ratio 4; feeding time 30 s)

Sample	HZSM-5	1-ZSM-5-im	2-ZSM-5-im	3-ZSM-5-im
Zn content of ZSM-5 zeolite, wt %	0	0.29	0.61	0.81
Yield of products, wt %				
Dry gas	1.8	2.2	2.0	2.0
Liquefied hydrocarbon gas, including:	26.1	27.7	26.2	26.7
isobutane	6.6	7.4	6.9	7.0
C ₃ –C ₄ olefins	17.8	17.3	16.8	16.9
propylene	8.4	8.9	8.4	8.4
Gasoline (s.b.–216°C)	45.9	45.4	45.5	47.6
Light gasoil (216–350°C)	15.3	14.8	15.0	13.1
Heavy gasoil (350°C–e.b.)	5.7	4.8	5.7	5.1
Coke	5.2	5.3	5.5	5.5
Conversion, wt %	79.0	80.5	79.2	81.8
Hydrogen transfer coefficient	0.894	1.024	0.963	0.978

increase in the concentration of acid sites in the samples compared to the unmodified zeolite.

The highest gasoline yield (48.4 wt %) is reached with 3-HZSM-5-ie zeolite, because specifically this sample is characterized by the highest concentration of acid sites, determined by TPD–NH₃ (Fig. 4).

Analysis of the liquid cracking products shows that introduction of 0.86 and 0.81 wt % zinc into ZSM-5 zeolite leads to an increase in the fraction of aromatic

hydrocarbons in the liquid cracking products from 35.1 to 38.9 and 37.6 wt %, respectively (Fig. 5).

Modification of zeolite with zinc (especially by ion exchange) probably leads to an increase in the amount of acid sites favoring the intermolecular hydrogen transfer, as indicated by an increase in the hydrogen transfer coefficient. The occurrence of such reactions is characterized by a decrease in the yield of unsaturated hydrocarbons in gaseous products (Tables 2, 3). However, a decrease in the fraction of olefins in liquid cracking

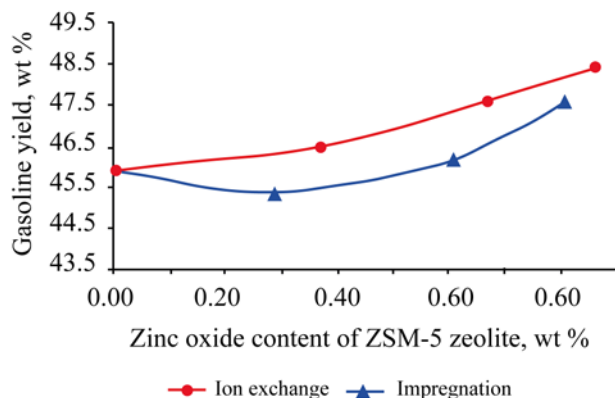


Fig. 4. Yield of the gasoline fraction in cracking of hydrotreated vacuum gas oil as a function of the ZnO content of ZSM-5 zeolite and of the zinc introduction method.

products is accompanied by an increase in the yield of aromatic compounds. Presumably, such changes in the group composition can lead to an increase in the research octane number by 0.5–1 unit.

CONCLUSIONS

We have studied how the zinc content of ZSM-5 zeolite and the procedure for zinc introduction influence

the physicochemical characteristics of the zeolite and the yield of aromatic hydrocarbons in cracking of hydrotreated vacuum gasoil. An increase in the content of zinc oxide introduced into ZSM-5 by impregnation to 0.81 wt % leads to a 10% decrease in the specific surface area and specific micropore volume, probably because of blocking of micropores with $(\text{ZnO})_n$, and to a 17% increase in the concentration of acid sites. Introduction of zinc by ion exchange, on the contrary, does not lead to noticeable changes in the textural characteristics, because zinc occupies cationic vacancies in the zeolite lattice, whereas the concentration of acid sites increases by a factor of more than 1.5 compared to unmodified ZSM-5. An increase in the acidity of zinc-modified zeolite may be due to preservation of the zeolite crystal structure and to the formation of $-\text{Al}-\text{O}-\text{Zn}-\text{O}-\text{H}$ sites.

Introduction of additives containing ZSM-5 zeolites modified with different amounts of zinc leads to an increase in the content of aromatic hydrocarbons in liquid cracking products by more than 2 wt %. Because of high concentration of acid sites in ZSM-5 zeolite modified with zinc by ion exchange, the use of this sample as a component of a catalyst for cracking of hydrotreated vacuum gasoil leads to an increase in the feed conversion from 79.0 to 81.2 wt %, in the hydrogen transfer coefficient from 0.894 to 1.199, in the gasoline yield

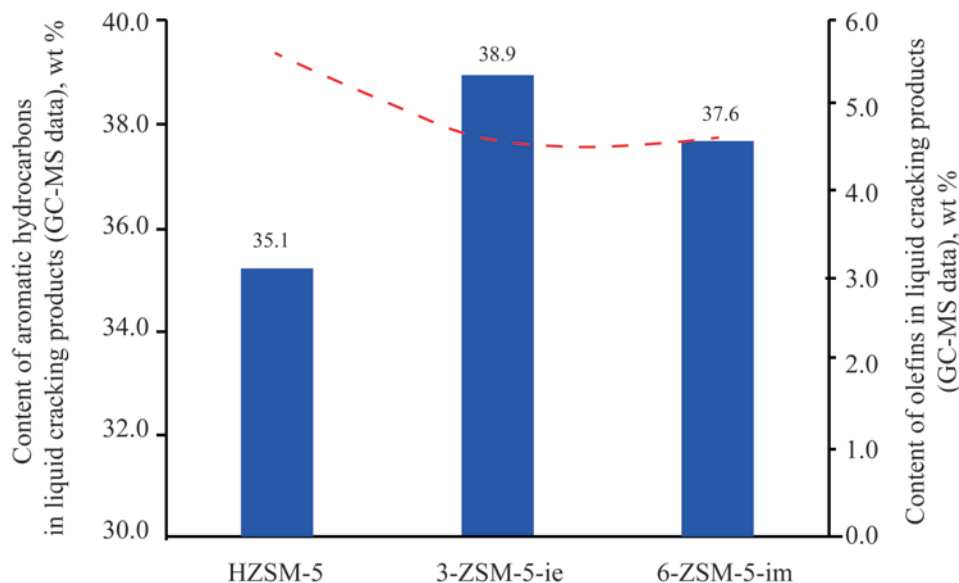


Fig. 5. Influence of the procedure for zinc introduction in ZSM-5 zeolite on the yield of aromatic hydrocarbons and olefins in cracking of hydrotreated vacuum gas oil (ZnO content of ZSM-5 zeolite 0, 0.86, and 0.81 wt %, respectively).

from 45.9 to 48.4 wt %, and in the content of aromatic compounds in the gasoline from 35.1 to 38.9 wt %.

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CONFLICT OF INTEREST

The authors declare no conflict of interest requiring disclosure in this article.

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