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Multifunctional Zeolite Based Catalysts for Tandem Reactions in Biomass Conversion

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Zeolites are widely used as catalysts or supports for catalysts in the processing of biomass waste into biofuels and value-added chemicals. Unlike chemicals derived from petroleum, molecules derived from biomass usually contain a large number of oxygen-containing groups, which makes their catalytic transformations even more complex. The development of multifunctional nanostructured zeolite-based catalysts and an understanding of their complex relationships between structure and properties are essential for successful cascade catalytic reactions of biomass processing. A cascade (or tandem) reaction is realized when several reaction stages are performed within a one-step process, which require different catalysts. For these reactions to be successful, two- or multi-functional catalysts or mixtures of catalysts are required at each reaction stage. In this review article, the main design factors of the zeolite-based catalyst that affect the structure–properties ratio, which may vary depending on the type of catalyst, are discussed. The main factors include the number and strength of acidic and basic sites, the interaction between them and metal sites, synergistic effects, the size and morphology of nanoparticles (NPs), nanostructures, porosity, etc.

1. Introduction

In recent decades, various types of biomass have become promising, environmentally friendly raw materials capable of replacing non-renewable fossil resources. The main goal of developing multifunctional zeolite-based catalysts suitable for cascade reactions is to control selectivity to prevent high energy and labour costs associated with separation and purification. This requires catalysts with a well-defined structure of active sites and a certain porosity (Gao et al., 2022). An analysis of the review articles published to date shows that zeolite-based catalysts demonstrate outstanding potential for future industrial applications in biomass processing due to their unique BAS/LAS ratio (Bronsted acid sites /Lewis acid sites) and multifunctional active centres (Li et al., 2022b). Zeolites are widely used as catalysts or substrates for catalysts in the processing of biomass waste into biofuels and value-added chemicals (Ennaert et al., 2016). Unlike chemicals derived from petroleum, molecules derived from biomass usually contain a large number of oxygen-containing groups, which makes their catalytic transformations even more complex. The development of multifunctional nanostructured catalysts based on zeolites and the understanding of their complex relationships between structure and properties are necessary for successful cascade catalytic reactions of biomass processing. Due to their high specific surface area, zeolites are suitable substrates for stabilizing nanoparticles of noble and earthy metals and metal oxides (Zhai et al., 2023). The combination of acidic properties of zeolites and redox properties of metals leads to the development of new universal catalysts with increased activity and selectivity. Metal nanoparticles catalyse processes involving hydrogen, including hydrogen activation, hydrogenation, and hydrogen transfer, while acid centres catalyse hydrolysis, dehydration, ring opening, etc. Several aspects of the structure of zeolite-based catalysts and their effect on catalysis will be discussed. This review allows the key factors of zeolite-based catalysts affecting the cascade reactions of biomass waste processing to be identified. This will help the researchers to design novel effective catalytic systems for the production of platform chemicals and fuels through the processing of biomass waste.

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2. Types of zeolites

Zeolite is a natural aluminosilicate with well-ordered pores and a crystalline structure (Krol, 2020). Synthetic zeolites, also called molecular sieves, have been widely used for many years as absorbents, catalysts, substrates for composite materials, etc. (Yang et al., 2021). Zeolite structures contain rings of different sizes that determine porosity. In addition to well-defined pore systems with different pore sizes, zeolites have acidic active Bronsted and Lewis centres, which allows for the production of various products in acid-base catalytic reactions. They also have high thermal and chemical stability, which makes them good candidates as substrates for multifunctional catalysts for biomass processing (Yang et al., 2021).

The three main types of zeolites include MFI (ZSM-5 and HZSM-5) with 10-membered rings, FAU, formed by 12-membered annular channels, and BEA (β), also containing 12-membered annular channels of different sizes. MFI zeolites are characterized by the presence of two interconnected channel structures, including pentasyl rings with medium pore sizes (Shamzhy et al., 2021). FAU contain larger pores (Li et al., 2022a). The pores of BEA are smaller than those of FAU, but larger than those of MFI (Bok et al., 2020). The pore size determines the diffusion of reacting molecules or the filtration of intermediates to control the reaction result. Depending on the Al/Si ratio (Al provides negative changes and acidity) The BAS/LAS ratio can vary, thus controlling the catalytic properties (Chassaing et al., 2018). In addition to the classical zeolites discussed above, new varieties have been developed, such as mesoporous (Singh et al., 2021) and hierarchical zeolites (Tan et al., 2022). It is worth noting that zeolites can also be alkaline when they undergo ion exchange with alkaline cations or when they contain inclusions of alkali metals (Yamamoto et al., 2018).

2.1 Si/Al molar ratio in zeolites

Changing the BAS/LAS ratio in zeolites can be achieved by changing the Si/Al ratio (Gao et al., 2022). The initial Si/Al ratio is created during the formation of an aluminosilicate framework, which can be influenced at the stage of gel formation (Lei et al., 2020) or during subsequent synthesis (Li et al., 2023). Desilication, i.e. the removal of certain types of silicon, leads to an increase in acidity due to BAS (with a counterion). The strength of the BAS is higher if they are isolated from each other. On the other hand, the use of dealumination (removal of aluminium) can reduce the acidity of the zeolite. For example, Li et al. (2016) catalysts based on dealuminated BEA zeolites have been reported to be effective in the cascade reaction of glucose conversion to 5-(ethoxymethyl)furfural (EMF). It is worth noting that the Si/Al ratio in zeolites can also affect the balance of hydrophilicity/hydrophobicity, stability, and how the catalyst works in catalytic processes (Moura et al., 2022). Zeolite MCM-22 and its layered modifications MCM-36 and ITQ-2 with different molar Si/Al ratios were synthesized and tested as catalysts for the dehydration of methanol and ethanol. The catalytic activity of the zeolite samples studied in the alcohol dehydration reaction depended mainly on the concentration of acid centres on the surface. It has been shown that an increase in the aluminium content in zeolite frameworks (a decrease in the Si/Al ratio) leads to an increase in the proportion of stronger acid centres, an increase in the activity of the catalyst during the dehydration of alcohols, and an increase in selectivity for esters (Marosz et al., 2020). Acid-catalysed aldol condensation between furfural and levulinic acid is presented in this work as an alternative for the production of C10 chemicals (two products) from renewable base molecules. Various zeolites were tested, and the Si/Al ratio of 23 and 80 was varied for ZSM-5. The best results were obtained when using ZSM-5 at a temperature of 423 K and an LA/FAL (Levulinic acid/Furfural) ratio of 2:1, with a yield of 50 % of the product and an almost insignificant number of adverse reactions (carbon balance after 24 hours: 91 %), while good behaviour was observed after three cycles of reuse without using any regeneration (Magyarová, et al., 2023).

The effect of the Si/Al ratio in zeolite Y on the efficiency of NiW catalysts for the second stage of hydrocracking was studied. The observed decrease in the concentration of Bronsted and Lewis acid centres is associated with an increase in the Si/Al ratio in zeolites. The catalysts were tested during hydrocracking of model (squalane) and real raw materials (unconverted petroleum (UCP) from a two-stage industrial hydrocracking plant) at various temperatures. It was found that an increase in the Si/Al ratio in zeolites leads to a decrease in catalytic activity and an increase in selectivity with respect to medium distillates (Golubev et al., 2021).

The most impressive effect of the Si/Al ratio was demonstrated in ref (Gao et al., 2022) for Pd nanoparticles deposited on H-ZSM-5 and used in the conversion of biologically based furfural (FAL) to cyclopentatone (CPO). Here, the Si/Al ratio tested was 25, 60, 130, and 200. It was demonstrated that an increase in the Si/Al ratio leads to a decrease in the number and strength of acid centres (both BAS and LAS), which leads to the best catalytic efficiency (98% selectivity with respect to CPO).

3. Incorporation of metal NPs

As discussed above, the conversion of biomass into chemicals and fuels often occurs during several reactions, each of which requires its own active center. Many reactions require the dual action of metal and acid centers

(Li et al., 2022b). Catalysts containing metal NPs and based on zeolites can be precisely tuned using a combination of different active centers to carry out a multi-stage reaction in a single reactor. Nanoparticles of noble metals embedded in zeolites (Pd (Gao et al., 2022), Pt (Li et al., 2022b), Ru (Chen et al., 2021)) exhibit high activity and selectivity, while nanoparticles of metals or metal alloys contained in large quantities on earth make it possible to find cheaper and at the same time effective alternatives (Co (Zhang et al., 2024), Ni (Yu et al., 2021), Ni-Pd (Zhang and Zhao, 2016)). The advantages of the redox properties of metallic nanoparticles, along with the acidic properties of zeolites, make it possible to develop universal catalysts with increased activity and selectivity.

The influence of the size of metal nanoparticles is often crucial in such catalysts, as it controls the number of active sites associated with the electronic structure and geometry of nanostructured metals (Wang and Lu, 2020). The NP size then changes the concentration of defects on the surface of the nanoparticles, thus affecting regent adsorption, cleavage, and bond formation. For example, the incorporation of Cu into Cu-BEA, developed for the oxidative cleavage of 1-phenyl-1,2-ethanediol (a model reaction of biomass processing), changes the properties of BAS and LAS, which leads to the production of the target product (Wang et al., 2022). In most cases, small NPs provide higher activity due to an increase in the number of active sites and defects (Li et al., 2022b). In addition to the size of the NPs, the structure of the NPs is an important parameter determining the catalytic properties. Catalysts based on HZSM-5 with different ends were tested in a cascade reaction consisting of dehydroaromatisation of limonene terpene and hydrodeoxygenation of stearic acid (Zhang and Zhao, 2016). During this cascade reaction, the terpene is first converted to p-cymole, with the simultaneous formation of H_2. After that, long-chain alkanes (C_{17} and C_{18}) are formed during a single hydrodeoxygenation of stearic acid. Analysis of various catalysts showed that bimetallic Pd-Ni/HZSM-5 is the best, while monometallic catalysts Ni or Pd showed low efficiency, i.e. low stearic acid conversion. The authors determined that dehydroaromatisation of limonene is activated by Pd active sites, while hydrodeoxygenation of stearic acid is catalyzed by Ni. It is noteworthy that the acid centers of HZSM-5, which can be changed by changing the Si/Al ratio, affect the catalytic properties of Pd-Ni. In addition, the corresponding porosity of the zeolite inhibits the condensation of limonene (a side reaction). However, another factor here is the enhanced hydrogen transfer between the two metals on the surface of HZSM-5, which improves the hydrodeoxygenation of stearic acid. Thus, both stages of these catalytic reactions are serviced by bimetallic zeolite-based catalysts.

Another bimetallic catalyst, Co/Sn-BEA, containing metal LAS, is used for selective hydrodeoxygenation of 5-Hydroxymethylfurfural (HMF) in 2,5-Dimethylfuran (DMF). (Zhang et al., 2024) In this catalyst, Sn-BEA nanocrystals (30-100 nm in size with a high Sn content) include Co NPs (3-10 nm in diameter) in a zeolite structure containing a lot of LAS. An analysis of the relationship between structure and properties showed that the concentrations of Co and Sn in Co/Sn-BEA are closely related to the catalytic properties. The role of Co nanoparticles is mainly to control the conversion of HMF, while isolated forms of Sn mainly affect the selectivity of the target product. Thus, smart control of the Co and Sn content in the catalyst can effectively regulate conversion and selectivity.

The reaction of LA with 1,4-pentanediol (1,4-PDO) in one reactor using Cu-Ni alloy NPs modified with Zn and deposited on H-ZSM-5 (Cu-Ni-Zn/H-ZSM-5) has been reported by Karanwal et al. (2021). This catalyst made it possible to obtain PDO with a yield of 93.4 % under optimal conditions. The addition of Zn limited the size of Cu-Ni nanoparticles to 4-8 nm, which made it possible to reduce the size of these nanoparticles during hydrogenation compared to the original Cu-Ni alloy nanoparticles. A large amount of LAS in Cu-Ni-Zn/ZSM-5 promotes LA adsorption, while conversion to γ-Valerolactone (GVL) occurs on the BAS of H-ZSM-5. LA hydrogenation was performed on Cu-Ni alloy sites. Further ring opening and hydrogenation led to the formation of PDO, which was facilitated by the spillover of H_2 into Cu-Ni alloy NPs doped with Zn.

4. Porosity of zeolites

For multifunctional zeolite-based catalysts, the pore size of zeolites plays an important role in cascade reactions, the course of which can vary due to different mass transfer of reagents and products of different sizes (Barakov et al., 2024).

Different types of zeolites have different pore sizes and structures (Zeng et.al., 2025). Both parameters are crucial for mass transfer of reacting molecules, intermediates, and target products, thus providing selectivity for certain substances, allowing intermediates to reach reactive sites or be filtered out (Mathew et al., 2024).

Another step in improving or modifying the reaction results is to obtain mesoporous (2-50 nm pore diameter) or hierarchical zeolites, i.e. zeolites with both micro- and mesopores (or macropores >50 nm in diameter). There are many examples of multifunctional catalysts based on mesoporous or hierarchical zeolites, so the current paper will be considered only a few of them (Singh et.al., 2025). Bai et al. synthesized a meso-/microporous Sn/Al plate catalyst based on MFI with LAS (Sn and Al) and BAS (Al-O(H)–Si) (Bai et al., 2018). The preferred catalytic reduction was the conversion of glucose to EMF, which is a three-step reaction. The first step is the isomerization of glucose to fructose, performed on Sn LAS, followed by the dehydration of fructose to 5-HMF. The last stage is the esterification of 5-HMF with ethanol to form an EMF based on Al–O(H)–Si. Crosstalk between several acid centres in a separate zeolite contributes to the cascade reaction of carbohydrate conversion in a single reactor. The double porosity of the above-mentioned zeolite ensures efficient mass transfer and facilitates the course of the reaction stages.

The single-stage conversion of cellulose to methyl lactate and methyl-2-methoxypropane using a bifunctional catalyst - Zn-containing FAU(Y) modified with Ga - was carried out in supercritical methanol (scMeOH) (Verma et al., 2017). This zeolite, was chosen because of the combination of micropores and large mesopores (10-30 nm), which provide a high specific surface area and provide easy access for bulk molecules such as cellulose and lignocellulose. Ga-ZnO nanoparticles were 3-7 nm in size and were well dispersed in Zn-containing FAU(Y). The increase in the amount of LAS and the decrease in the BAS content was explained by the addition of Ga to ZnO. Optimal acidity along with a high surface area ensured the selectivity of cellulose conversion. Under optimal conditions, methyl acrylate and methyl-2-methoxypropionate were obtained in a yield of 70.6 % with the addition of Ga 1.8 wt. % and Zn 9.8 wt. %.

Other examples of zeolite-based catalysts with hierarchical porosity include Zr-Al-BEA (Song et al., 2017) for converting FAL to γ-valerolactone (GVL), ZrP catalysts for converting biomass-derived FAL to isopropyllacetate and GVL (Ye et al., 2020), as well as FAU-based catalysts for pre-esterification from bio-oil (Osatiashtiani et al., 2017). Tan et al. (2022) developed acid-base catalysis based on SAPO-11 with different Si/Al ratios and Zr-SBA-15 (mesoporous framework) with different Si/Zr ratios. In combination with Raney Ni, these catalysts were effective in converting phenol during a cascade reaction simulating the pyrolysis transformation of oil (Tang et al., 2021).

Cho et al. (2020) used an original strategy for encapsulating Pt nanoparticles (with an average diameter of 6 nm) using a cationic polymer, poly (diallyldimethylammonium chloride) (PDDA), in MFI and BEA zeolites. These catalysts have shown high efficiency in regulating the sequence of interactions of reagents and intermediates with various types of active sites. It has been found that changing the pore size of zeolite is an effective way to regulate selectivity in cascade reactions with large-sized intermediates. Pt nanoparticles encapsulated in H-BEA (0.75 nm pore size) (Pt@H-BEA) can catalyze the single-stage conversion of CPO obtained from biomass into cyclic hydrocarbons C_{10} (bicyclopentane and decalin) with a total yield of 78 %. On the other hand, Pt nanoparticles encapsulated in MFI with a micropore size of 0.56 nm and a similar density of metallic and acid centres give mainly cyclopentane (~70 %). Kinetic analysis showed that the difference in pore size between these two zeolites is the main reason for the dramatic change in selectivity, probably due to a higher energy barrier for the formation and diffusion of larger molecules. Pt-NPs deposited (not encapsulated) on H-BEA or H-ZSM-5 are not selective. In relation to cyclic hydrocarbons, C_{10} is used in CPO conversion, which underlines the importance of the composite catalyst architecture.

Ru NP-containing HZSM-5 catalysts had a relatively high selectivity in hydrogenolysis (Zheng et al., 2018). In this work, three HZSM-5 zeolites of cross-shaped, spherical, and cuboid shape with a similar Si/Al ratio were studied in the development of Ru catalysts for the hydrogenolysis of guaiacol to benzene, which proceeds in a cascade reaction converting guaiacol to phenol, and then phenol to benzene. For all three matrices, the Ru content was ~5 wt. %, which led to particle sizes in the range of 4-5 nm. The authors found that for cross-shaped HZSM-5, benzene was obtained in 97 % yield. The other two substrates did not show selectivity. It was found that the cross-shaped HZSM-5 has a high mesoporosity and makes it possible to obtain well-defined nanoparticles with a narrow particle size distribution. Moreover, the interaction between Ru and HZSM-5 nanoparticles leads to the formation of two types of Ru: electron-deficient and electronegative.

Biocrude oil hydrodeoxygenation was performed using a Ru NPs based on four zeolites – BEA, HZSM-5, HY, and SAPO-34, the pores of which differ in size and shape (Yan et al., 2020). Ru catalysts based on BEA and HY showed a higher MP yield than that of HZSM-5. This is consistent with the fact that the major porn channels in BEA and HY consist of 12 x 12 rings compared to channels in HZSM-5 (10x10 rings). The SAPO-34-based catalyst turned out to be the worst, which can be explained by its smaller size due to the eight-membered ring. A similar result was obtained by other authors in the reaction of C6 sugars to FAL, where the least selective catalyst was a catalyst based on HZSM-5 with smaller pores (Gürbüz et al., 2013). The above data demonstrate that mass transfer (diffusion), which depends on the morphology of the zeolite, is very important in many catalytic (and especially cascade) reactions (Chen et al., 2021).

5. Conclusions

The data presented in this review article clearly shows that cascade reactions in biomass waste processing require dual or multifunctional catalysts to optimize each stage of the process and enable the reaction to take place in a single reactor. Depending on the raw materials and target products, it is necessary to manufacture various catalysts, taking into account their stability, acidity, interaction with metals, porosity, etc. For any catalyst, the ratio of alkali to alkalinity and strength are crucial. In zeolites, they can be adjusted by changing the Si/Al ratio during synthesis or subsequent processing by desulfurization, dealumination, introduction of other components, etc. In the presence of metals, their interaction with acid-base centres often leads to synergy, which leads to an intensification of reactions or an increase in selectivity. Separation of the active centres is another important way to improve the catalyst, allowing to maintain the integrity of the active centres. The main advantages of zeolite supports are product quality control by pore size and shape, as well as ease of acidity regulation.

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