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SYNTESIS AND PROPERTIES OF Ti-Si CATALYSTS FOR THE SELECTIVE OXIDATION OF ALKENES TO KETONES WITH HYDROGEN PEROXIDE

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Abstract:*This paper presents the results of the study of heterogeneous catalysts based on Ti–Si for the selective oxidation of organic compounds, employing hydrogen peroxide as a green oxidizing agent. The catalysts were synthesized using a hydrothermal sol–gel method, carefully controlled to ensure the effective integration of titanium species into the silica matrix, resulting in a sophisticated porous framework. The characterization of the materials' physicochemical and structural properties involved various analytical techniques such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), UV–Vis diffuse reflectance spectroscopy (UV–Vis DRS), nitrogen adsorption–desorption analysis (BET), as well as scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The findings confirmed the formation of a TS-1 type structure with high crystallinity, significant surface area, and notable porosity. The presence of an FT-IR absorption band at approximately ~960 cm^{-1} and a UV–Vis absorption peak near ~210 nm provided evidence of successful incorporation of isolated tetrahedrally coordinated Ti^{4+} ions within the silica framework, enhancing catalytic performance. The efficiency of the Ti–Si catalysts in the selective oxidation of organic compounds using hydrogen peroxide was assessed, focusing on the conversion of butene-1 to methyl ethyl ketone (MEK) as a model reaction. Results demonstrated that the catalytic activity and selectivity were influenced by the concentration and dispersion of titanium in the silica matrix. An optimal titanium loading level achieved a balance between reaction rates and selectivity, while excessive titanium concentrations led to the formation of additional species within the framework and partial degradation of hydrogen peroxide. Moreover, these catalysts exhibited remarkable stability and reusability over multiple cycles, indicating strong structural integrity and resistance to deactivation. The combination of a large surface area, porous structure, and well-dispersed active Ti sites significantly improved catalytic efficiency. The results of research confirmed the potential of Ti–Si heterogeneous catalysts as effective materials for sustainable selective oxidation processes employing hydrogen peroxide.*
Keywords: *Ti–Si catalysts, TS-1 zeolite, hydrogen peroxide, selective oxidation, heterogeneous catalysis, hydrothermal sol–gel method, butene-1 oxidation, methyl ethyl ketone (MEK), titanium dispersion, porous materials.*

İNTRODUCTION

In recent years, in the processes of oxidation of organic compounds, a tendency has been observed to replace expensive and toxic oxidants with environmentally friendly oxidants – molecular oxygen or hydrogen peroxide. The most preferable is the use of H_2O_2, which is associated with the lower cost of technological equipment compared to oxidation with oxygen, since the oxidation process with oxygen is carried out at elevated temperatures and pressures. On the other hand, oxidation with hydrogen peroxide is less dangerous [1-5].

In oxidation processes, complexes of transition metals are mainly used as catalysts; however, homogeneous catalysts for selective oxidation, as a rule, possess

high activity and selectivity, but are characterized by low productivity associated with the degradation of the complex during the catalytic process, as well as by problems related to the separation of the catalyst from the reaction products and its regeneration [6-9].

One of the main tasks of liquid-phase oxidative catalysis is the creation of solid-phase catalysts that possess high activity and selectivity and at the same time are stable to the influence of the reaction medium. One of the possible ways to solve this problem is the introduction of transition metal ions or their complexes into the framework or pores of mesoporous materials, where the active centers are in an isolated state and are homogeneously distributed in the solid matrix [10-11].

Significant success in this direction was achieved by the Italian company Enichem, which in 1983 developed a microporous titanium silicate catalyst TS-1 for liquid-phase oxidative catalysis. This catalyst has the crystalline structure of silicalite ZSM-5, in which part of the silicon atoms is isomorphously substituted by titanium ions. At present, TS-1 is considered the best heterogeneous catalyst for the liquid-phase oxidation of various classes of organic compounds with an aqueous solution of hydrogen peroxide. The process of phenol oxidation with hydrogen peroxide over TS-1 has found practical industrial application – with its help, 10,000 tons of hydroquinone and a large amount of pyrocatechin are produced per year. The main disadvantage of microporous titanium silicates is the small pore size, which makes their use in oxidation reactions of large molecules impossible [12-13].

At present, the number of works on the synthesis, study, and application in oxidative catalysis of new materials – mesoporous silicates containing ions of various transition metals, in particular titanium – is increasing. The specific surface area of such materials reaches 1000 m^2/g. Under conditions of liquid-phase catalysis, large organic molecules can freely penetrate into the pores of these materials, unlike microporous materials. The development of such materials is very important for solving the problems of oxidative catalysis using an aqueous solution of hydrogen peroxide [14-15].

Objective of the study is syntesis of mesoporous titanium silicate catalysts, the study of physicochemical properties, and their catalytic properties in the liquid-phase oxidation reaction of alkenes to ketones.

EXPERİMENTAL PART

Tetraethyl orthosilicate (TEOS, $Si(OC_2H_5)_4$) and titanium isopropoxide (TTIP, $Ti(OCH(CH_3)_2)_4$) were employed as the silicon and titanium precursors, respectively, in the synthesis process. Hydrogen peroxide (H_2O_2, 30% weight) was utilized as the oxidizing agent. The experimental procedure involved the use of deionized water, ethanol (C_2H_5OH), and ammonia solution ($NH_3 \cdot H_2O$). All chemicals utilized were of analytical grade and underwent no additional purification steps.

Porous Ti–Si catalysts were synthesized through the hydrothermal sol–gel technique. Initially, TEOS was dissolved in ethanol with continuous stirring to achieve a homogeneous solution. Subsequently, titanium isopropoxide was added dropwise with precise control to ensure uniform integration of titanium species within the silica matrix. The molar ratio of the synthesis mixture was adjusted to SiO_2: TiO_2: H_2O: EtOH = 1: (0.01–0.05): 20: 10. The hydrolysis and condensation reactions were initiated by the gradual addition of deionized water and ammonia solution. The resulting mixture was stirred for a duration of four to six hours until gel formation occurred. Following

gelation, the material underwent hydrothermal treatment at 150 °C for 24 hours in a Teflon-lined autoclave following a 12-hour aging period at room temperature.

After crystallization, the solid was filtered, washed thoroughly with ethanol and deionized water, and subsequently dried overnight at 100 °C. A subsequent calcination process in air at 550 °C for five hours was conducted to remove organic residues and stabilize the Ti–Si structure.

The crystallinity and phase composition of the catalysts were analyzed using X-ray diffraction (XRD) with Cu Kα radiation (λ = 1.5406 Å) within the 2θ range of 5–80°. Fourier-transform infrared spectroscopy (FT-IR) was employed to identify functional groups and Ti–O–Si bonds through infrared spectra recorded within the 400–4000 cm^{-1} range using the KBr pellet method.

Evaluation of the specific surface area, pore volume, and pore size distribution of the catalysts was performed through nitrogen adsorption–desorption analysis at 77 K using a surface area analyzer. The BET method was utilized for surface area determination, while the BJH method was employed for assessing pore size distribution.

Morphological analysis and particle size characterization of the catalysts were carried out using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Furthermore, UV–Vis diffuse reflectance spectroscopy (UV–Vis DRS) measurements were conducted within the 200–800 nm range to ascertain the coordination state of titanium species using a UV–Vis spectrophotometer.

The catalytic performance of the synthesized materials was evaluated through the selective oxidation of organic compounds like phenol and cyclohexene. A standard catalytic test involved dispersing 0.05 g of catalyst in a 25 mL reaction mixture containing the substrate and solvent, followed by stirring at 60 °C. Controlled addition of hydrogen peroxide was carried out to regulate the reaction rate and prevent rapid decomposition, allowing the reaction to proceed for four hours under atmospheric pressure.

The catalyst was separated using filtration or magnetic separation, and the reaction products were analyzed using gas chromatography (GC-FID). Conversion, selectivity, and yield were quantified using the following formulas:

$X(\%) = [(n_o - n)/n_o] \times 100$

$S(\%) = [ndesired/(n_o - n)] \times 100$

$Y(\%) = [ndesired/n_o] \times 100$

RESULTS AND DISCUSSION

The hydrothermal sol-gel synthesis technique was found to be a successful method for producing Ti-Si catalysts with intricate porous structures, high surface area, and uniform pore distribution. Through the examination of X-ray diffraction (XRD) patterns (fig.1), the presence of crystalline TiO_2 phases was notably absent, indicating the successful integration of titanium components into the silica framework, leading to the creation of a traditional silicalite configuration. The dispersion of titanium throughout the amorphous silica matrix appeared to be extensive, preventing the formation of distinct TiO_2 phases, a fact supported by the broad diffraction peaks and the lack of anatase reflections. The absence of anatase signals suggests that titanium is predominantly present in a finely dispersed form within the framework.

The X-ray diffraction (XRD) analysis of the synthesized TS-1 type catalyst serves as a critical method for elucidating its structural properties and validating the successful

creation of the desired titanium silicalite framework. The XRD pattern obtained within the 2θ range of approximately 10–80° reveals a distinct series of well-defined and sharp peaks, indicating a high level of crystallinity in the material. Notably, the identifiable reflections observed at approximately 2θ ≈ 7–9° and 23–25° are characteristic of the MFI-type zeolite structure, fundamental to TS-1 materials. The presence of these peaks distinctly indicates that the hydrothermal sol-gel synthesis procedure resulted in the formation of an ordered silicalite lattice rather than an amorphous silica phase.

A significant aspect of the XRD data is the absence of diffraction peaks associated with crystalline titanium dioxide phases, particularly anatase, which typically appear at around 25°, 37°, and 48° in 2θ. This absence suggests that titanium species are not present as distinct TiO_2 particles but are instead evenly dispersed within the silica framework.

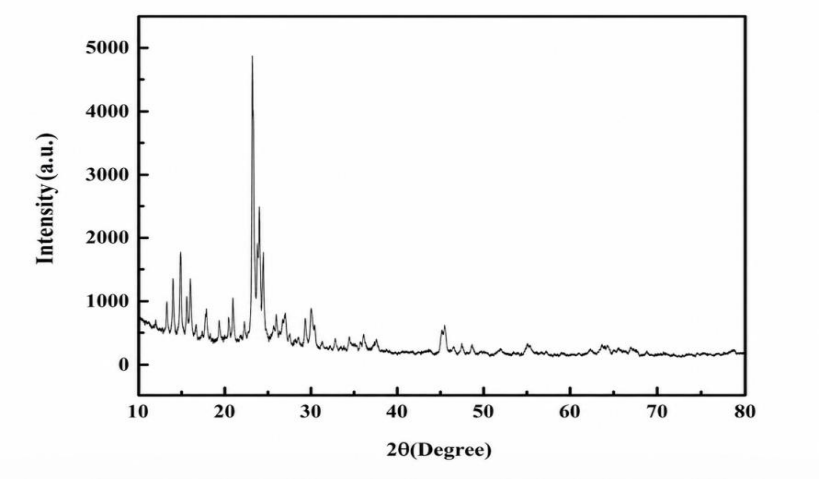

Fig. 1. XRD pattern of TS-1 catalyst

This implies successful integration of titanium atoms into the silicalite lattice structure as isolated Ti^{4+} sites, a crucial prerequisite for achieving enhanced catalytic activity in selective oxidation reactions. The uniform distribution of titanium throughout the framework hinders the formation of inactive bulk TiO_2 phases and boosts the catalyst's efficiency.

Furthermore, the high intensity and narrow width of the diffraction peaks indicate that the synthesized TS-1 catalyst possesses a well-established crystalline structure with minimal structural defects. This structural integrity is vital for ensuring uniform pore architecture and accessibility to active sites. Although minor background signals may be present across the entire diffraction spectrum, possibly originating from a small quantity of amorphous silica or structural irregularities, they have negligible impact on the overall crystallinity of the material.

The structural characteristics unveiled by the XRD investigation hold substantial catalytic relevance. The well-defined MFI framework, coupled with the absence of titanium species outside the framework, ensures the formation of isolated tetrahedrally coordinated Ti^{4+} centers within the silica matrix. These centers are pivotal for activating hydrogen peroxide and generating reactive titanium-peroxo intermediates (Ti–OOH), crucial for selective oxidation reactions like olefin epoxidation and aromatic hydroxylation. Consequently, the XRD outcomes not only validate the successful synthesis of TS-1 but also affirm its efficacy as a proficient heterogeneous catalyst.

In conclusion, the XRD examination signifies that the synthesized Ti-Si catalyst manifests a characteristic TS-1 crystalline structure, high phase purity, and effective integration of titanium into the silica framework, essential for achieving superior catalytic performance, selectivity, and durability in environmentally sustainable oxidation processes utilizing hydrogen peroxide. The successful integration of titanium into the silicate framework was corroborated by Fourier-transform infrared (FT-IR) spectra, where a distinctive absorption band at approximately 960 cm^{-1} indicated the presence of Ti–O–Si linkages, providing compelling evidence of titanium substitution for silicon in the framework.

The diagram in figure 2 displays the Fourier-transform infrared (FT-IR) spectra of TS-1 type titanium-silicate catalysts within the spectral range of 400-4000 cm^{-1}, providing crucial insights into the chemical composition, bonding arrangement, and incorporation of titanium components within the silica framework.

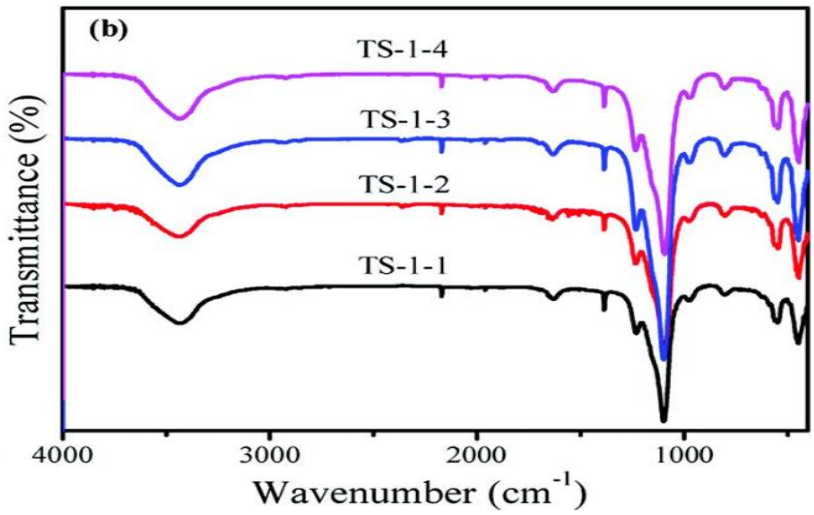

Fig. 2. FT-IR spectra of TS-1 catalysts (TS-1-1–TS-1-4)

The spectra from various specimens (TS-1-1, TS-1-2, TS-1-3, and TS-1-4) exhibit similar overall profiles, emphasizing the retention of the fundamental silicate structure across all samples synthesized through the hydrothermal sol-gel method.

A prominent and broad absorption peak observed at approximately 1100 cm^{-1} is associated with the asymmetric stretching vibrations of Si-O-Si bonds, confirming the establishment of the silica network structure. Additionally, the peak around 800 cm^{-1} corresponds to the symmetric stretching vibrations of Si-O bonds, while the signal at about 460 cm^{-1} is linked to the bending vibrations of the Si-O-Si linkage. The presence of these distinct peaks indicates the successful formation of a well-defined silicate structure rather than an amorphous phase.

The most noteworthy feature in the FT-IR spectra is the peak detected around 960 cm^{-1}, attributed to the stretching vibration of Ti-O-Si bonds, providing clear evidence of the effective integration of titanium species into the silica matrix. Variations in the intensity of this peak among different samples suggest differences in titanium content and dispersion, with a stronger peak indicating a higher degree of titanium integration into the framework and an increased presence of active Ti sites.

At higher wavenumbers, a wide absorption band at approximately 3400 cm^{-1} is observed, corresponding to the stretching vibrations of hydroxyl (-OH) groups and adsorbed water molecules on the catalyst surface. Furthermore, a weaker peak near 1630 cm^{-1} is attributed to the bending vibration of molecular water. These characteristics indicate the existence of surface hydroxyl groups and physically adsorbed water, which could impact adsorption properties and catalytic performance.

A comparative analysis of the spectra demonstrates that the Ti-O-Si peak strengthens from TS-1-1 to TS-1-4, indicating an increased integration and dispersion of titanium species within the silica framework. Meanwhile, the positions of the primary Si-O-Si peaks remain unchanged, indicating the preservation of the overall structural integrity of the silicate framework during synthesis.

From a catalytic perspective, these findings support the predominant presence of titanium in a framework position as isolated Ti^{4+} species, crucial for catalytic effectiveness in oxidation reactions. The formation of Ti-O-Si bonds facilitates the creation of active sites capable of interacting with hydrogen peroxide and initiating reactive intermediates. Consequently, the FT-IR analysis highlights the possession of essential structural characteristics by the synthesized TS-1 catalysts for efficient and selective catalytic oxidation processes.

The ultraviolet-visible diffuse reflectance spectra (UV-Vis DRS) of the synthesized TS-1 catalysts were examined in the wavelength range of 200-500 nm to investigate the coordination state, dispersion, and electronic configuration of titanium species incorporated into the silica framework.

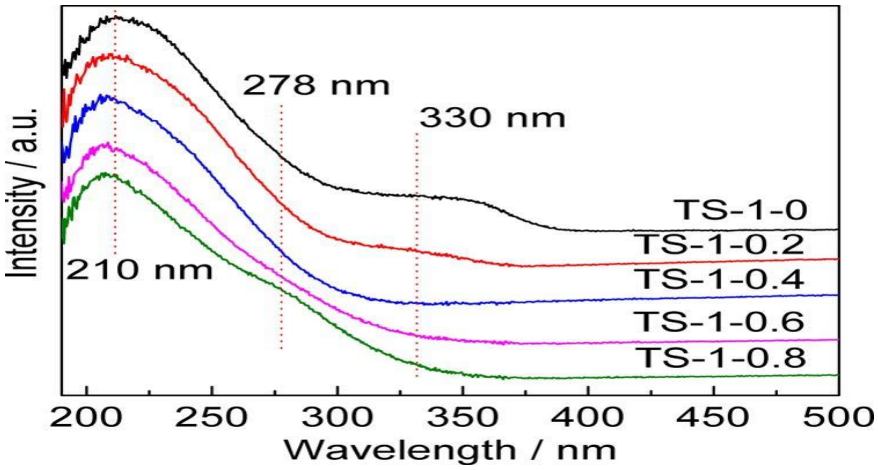

Fig. 3. UV–Vis DRS spectra of TS-1 catalysts

Analysis of the results unveiled distinctive absorption features in the ultraviolet region across all samples (TS-1-0, TS-1-0.2, TS-1-0.4, TS-1-0.6, and TS-1-0.8), which are directly associated with the different titanium species present in the catalysts (fig.3).

A significant absorption band, observed at approximately 210 nm, corresponds to the ligand-to-metal charge transfer transition of isolated tetrahedrally coordinated Ti^{4+} species within the silicalite framework. This absorption band serves as a crucial indicator of framework titanium, validating the successful substitution of titanium atoms within the SiO_2 lattice rather than the formation of distinct phases. Particularly noteworthy is the relatively intense nature of this band, particularly in samples with

lower titanium loading, indicating a high dispersion of isolated Ti species that act as the primary active sites in selective oxidation reactions.

Moreover, a broader absorption feature around 278 nm signifies partially polymerized or oligomeric titanium species, which are typically viewed as intermediary structures between isolated framework titanium and extra-framework titanium oxide clusters. The gradual enhancement in the intensity of this feature with increasing titanium content suggests that a portion of titanium species begins to aggregate as the titanium loading rises, leading to a slight decrease in dispersion.

Additionally, a faint but observable absorption tail extending towards 330 nm is detected in certain samples, indicating the existence of extra-framework TiO_2 species or small clusters resembling anatase. The escalation in the intensity of this feature with higher titanium content suggests that excessive titanium loading encourages the formation of less active or inactive titanium oxide species external to the silicate framework. This occurrence could be detrimental to catalytic applications as it reduces the population of isolated active Ti^{4+} centers, potentially leading to the non-selective decomposition of hydrogen peroxide. Comparative examination of the spectra demonstrates that samples with lower titanium content exhibit more pronounced absorption at 210 nm and weaker signals at higher wavelengths, highlighting a higher prevalence of isolated framework Ti species.

Conversely, samples with elevated titanium loading display increased absorption in the 278-330 nm range, indicating the formation of aggregated or extra-framework titanium species. This trend underscores the reduction in titanium dispersion as the loading exceeds an optimal threshold.

These findings bear significant implications from a catalytic standpoint. The presence of isolated tetrahedrally coordinated Ti^{4+} species facilitates the effective activation of hydrogen peroxide and the formation of reactive titanium-peroxo intermediates (Ti-OOH), crucial for selective oxidation reactions such as olefin epoxidation. Conversely, the formation of polymerized or bulk TiO_2 species may trigger undesirable side reactions, including non-selective oxidation and hydrogen peroxide decomposition.

To summarize, the UV-Vis DRS analysis confirms that the synthesized TS-1 catalysts predominantly host titanium in a tetrahedral coordination state at lower loading levels, while increased titanium content leads to partial aggregation and extra-framework species formation. These results underscore the critical importance of controlling titanium content to achieve optimal dispersion of active sites and enhance catalytic efficiency in environmentally friendly oxidation processes.

The nitrogen adsorption-desorption isotherms exhibited mesoporous structures characterized by typical type IV behavior with a hysteresis loop. Depending on the titanium content, the catalysts exhibited specific surface areas ranging from 700 to 1100 m^2/g and pore diameters between 2 and 5 nm, significantly influencing enhanced mass transfer and active site accessibility.

Microscopic analyses conducted through transmission electron microscopy (TEM) and scanning electron microscopy (SEM) unveiled the catalysts' uniform morphologies, nanoscale particle sizes, and well-defined porosity networks. The incorporation of magnetic nanoparticles into modified or magnetic composites facilitated efficient catalyst separation post-reaction. Alternatively, macroporosity arose from inter-crystalline voids between individual TS-1 building blocks assembled into larger

hierarchical structures, achievable through the crystallization of a macroscopically structured dry gel containing zeolite nanocrystals (fig.4).

This research investigates the catalytic efficiency of Ti–Si alloys in the selective oxidation of butene-1 to methyl ethyl ketone (MEK) utilizing hydrogen peroxide. Findings demonstrate a direct relationship between the titanium content and the conversion of the substrate, with optimal outcomes observed within a titanium loading range of 3–5%. However, excessive levels of titanium induce a decrease in selectivity attributed to the emergence of additional extra-framework Ti species and the partial degradation of hydrogen peroxide.

Significantly, the catalyst's performance is significantly influenced by the concentration and dispersion of titanium throughout the silica matrix. Increasing the titanium loading from 1% to 5% leads to heightened conversion rates while maintaining the preferential synthesis of desired compounds. A comprehensive summary of the catalytic results is presented in table 1.

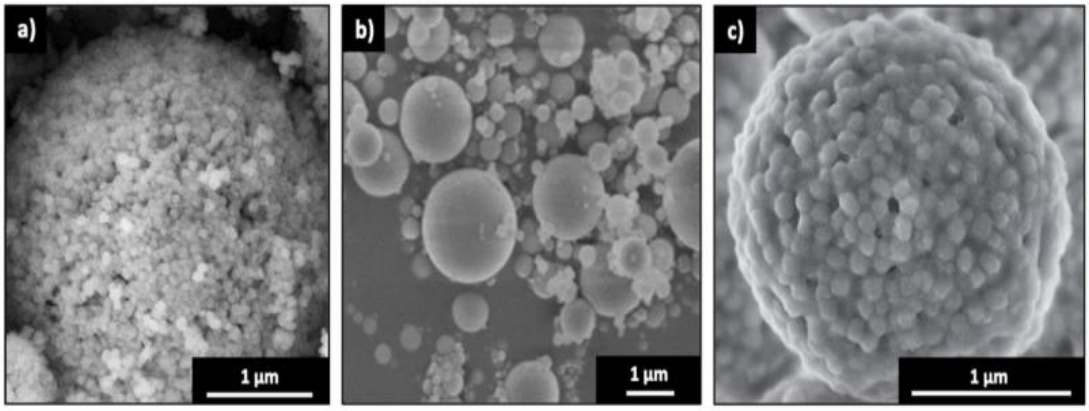

Fig. 4. SEM images of porous zeolite microspheres of silicalite-1 (a) and TS-1 (b–c) nanocrystals

Table 1

Catalytic research data

Catalyst	Conversion (%)	Selectivity (%)	Yield (%)
Ti–Si (1% Ti)	65	85	55
Ti–Si (3% Ti)	78	90	70
Ti–Si (5% Ti)	82	88	72

The evaluation of catalytic performance concerning titanium concentration is detailed in table 1. The optimal performance was observed with the Ti–Si catalyst containing 3% Ti, which also exhibited an ideal conversion-to-selectivity ratio of 78% to 90%. This underscores the necessity of a meticulous dispersion of individualized Ti entities to achieve sufficient catalytic effectiveness.

During catalysis, the generation of reactive titanium–peroxo species (Ti–OOH) takes place, where hydrogen peroxide is activated on isolated Ti sites. These species

subsequently provide oxygen to the organic substrate, reducing the formation of undesired byproducts and ensuring favorable selectivity under mild reaction conditions.

After five consecutive cycles, assessments of reusability indicated that the Ti–Si catalysts maintained robust catalytic performance with a minor reduction in conversion (<5%). This confirms the structural durability and resistance to catalyst deactivation, essential qualities for practical applications.

This the findings underscore the exceptional performance of Ti–Si-based heterogeneous catalysts in selectively oxidizing organic compounds using hydrogen peroxide. These materials present promising opportunities for oxidation processes beneficial to industry and the environment due to their high activity, specificity, and resilience.

CONCLUSION

Using hydrogen peroxide as a green oxidant, Ti–Si-based heterogeneous catalysts were effectively produced in this study using a hydrothermal sol–gel process and assessed in the selective oxidation of butene-1. The resulting catalysts showed homogeneous dispersion of titanium species within the silica framework, large surface area, and well-developed porous structures. Titanium was primarily found in isolated tetrahedrally coordinated Ti (IV) sites, which are in charge of catalytic activity, according to structural analysis.

The catalytic results showed that Ti–Si materials are highly active and selective in oxidation reaction of butene to MEK. Titanium content had a significant impact on catalytic performance; an ideal Ti loading of about 3% offered the optimum balance between conversion and selectivity. Because of the partial breakdown of hydrogen peroxide and the creation of extra-framework titanium species, a higher titanium content resulted in a small decrease in selectivity.

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