

Description**TITLE OF INVENTION: ETHYLENE PRODUCTION PROCESS THROUGH CATALYTIC DEHYDRATION REACTION OF ETHANOL USING HZSM-5 CATALYST IMPREGNATED WITH TUNGSTEN-CONTAINING COMPOUNDS**

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Field of the Invention

The present invention relates to the catalytic reaction of ethanol to ethylene. More specifically, the present invention relates to a process for producing ethylene by dehydration of ethanol using a HZSM-5 catalyst modified with a tungsten-containing compound.

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Background of the Invention

Ethylene, C_2H_4 , is an organic compound widely used as a raw material for making various chemicals and polymers, such as polyethylene, polyvinyl chloride, polystyrene, and ethylene oxide. In terms of its application, this compound is widely used in the agricultural, pharmaceutical, and plastic industries. Due to its wide range of applications, the annual production of ethylene at the global level is known to exceed the production of other organic compounds.

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The conventional method that currently is widely used for ethylene production is thermal cracking of long-chain hydrocarbons from fractional distillation of petroleum into ethylene. However, the raw material for this thermal cracking process is petroleum, which is a non-renewable natural resource gradually becoming increasingly rare and expensive. This process takes place at a temperature of 600-1000°C, hence consuming a lot of energy. Additionally, this process is known to generate significant CO₂ emissions, which contribute to global warming. Therefore, there is a need for an alternative ethylene production processes that avoid the problems of petroleum scarcity, high energy consumption (thus expensive production costs) and environmental issues (global warming) associated with

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the conventional process of ethylene production through thermal cracking.

One of the alternative processes for producing ethylene that is currently being researched and developed is the catalytic dehydration of ethanol into ethylene. This process employs ethanol derived from renewable biomass, which mitigates dependence on petroleum-based hydrocarbons, a non-renewable resource subject to depletion and cost fluctuations. As it does not use hydrocarbons from petroleum, this method has the potential to significantly reduce carbon emissions. In addition, because this method avoids the high temperatures associated with thermal cracking processes, it has the potential to be more economical by reducing energy consumption costs.

For the method of making ethylene through the catalytic dehydration reaction of ethanol into ethylene, extensive research and development has been done to find the most suitable type of catalyst for this catalytic reaction. One type of catalyst that has received much attention is zeolite, especially zeolite ZSM-5. The selection of this catalyst, especially in its acid form (HZSM-5), is based on its fairly good sorption and catalytic properties. At reaction temperatures above 300°C, the HZSM-5 catalyst is effective in producing ethylene with high selectivity. However, at this relatively high temperature, coking is prone to occur on the external and internal surfaces of HZSM-5, which causes catalyst deactivation and shortens the catalyst's life. On the other hand, at lower reaction temperatures, there is a decrease in the catalyst's performance to produce ethylene.

Several methods for producing ethylene by dehydration of ethanol using modified HZSM-5 have been disclosed. One of the methods is disclosed in Chinese Patent CN101274286A. In the Chinese Patent document CN101274286A, the HZSM-5 catalyst is modified by impregnating active metals into the

HZSM-5 catalyst with a ratio ranging from 0.5 to 7% w/w. The active metals used for impregnation include Ca, Fe, Zn, Mn, Zr, and La. Under operating conditions of atmospheric pressure and at a temperature range of 250-290°C, it is disclosed that the modified HZSM-5 catalyst was capable of achieving ethanol conversion of 96-99% with ethylene selectivity of 98-99%.

Although the production of ethylene through ethanol dehydration using the modified HZSM-5 catalyst disclosed in Chinese Patent CN101274286A is said to be able to increase the conversion of ethanol to ethylene as well as the selectivity of ethylene, the reaction temperature of ethanol dehydration to ethylene was still quite high, which was around 250-290°C. The high reaction temperature indicates that the dehydration reaction of ethanol to ethylene using the modified HZSM-5 disclosed in the Chinese Patent CN101274286A required relatively high energy consumption. Therefore, alternative modifications to the HZSM-5 catalyst are still needed to enable the modified HZSM-5 catalyst to catalyze the dehydration reaction of ethanol to ethylene at a lower reaction temperature while maintaining high ethanol conversion rates and ethylene selectivity.

United States Patent No. 4,698,452 discloses a process for producing C₂-C₄ olefins from ethanol using a ZSM-5 zeolite catalyst modified (impregnated) with Zn and Mn metals. However, the temperature of the process in this patent was still relatively high, which was around 300-450°C. Although this process was said to produce a fairly high ethanol conversion, its ethylene selectivity was relatively low.

United States Patent No. 4,847,223 discloses a process for converting aqueous ethanol to ethylene using HZSM-5 catalyst modified with trifluoromethanesulfonic acid (CF₃SO₃H). This conversion process was mentioned to take

place at a relatively low reaction temperature, namely 180-275 °C. However, the conversion of ethanol to hydrocarbons in this process was still relatively low, which was less than 60%.

5 Therefore, there is still a necessary to provide a process of making ethylene through ethanol catalytic reaction that can overcome the weaknesses mentioned above. One of the objectives of this invention is to solve the technical problems related to the performance of HZSM-5
10 catalysts in the process or method of making ethylene through ethanol dehydration. The main objective of the present invention is to provide a process or method for producing ethylene by dehydration of ethanol using a modified HZSM-5 catalyst showing a good performance at
15 relatively lower reaction temperatures. The other objective of the present invention is to provide a process or method for producing ethylene by dehydration of ethanol using a modified HZSM-5 catalyst to produce ethylene with a sufficiently high conversion rate and selectivity.
20 Another objective of the present invention is to produce ethylene from these processes.

Summary of the Invention

The present invention aims to overcome the problems
25 related to the production of ethylene through ethanol dehydration using HZSM-5 catalysts. This objective can be achieved through the process of producing ethylene through the catalytic dehydration reaction of ethanol provided according to the present invention. The process of
30 producing ethylene through the catalytic dehydration reaction of ethanol is achieved with the following steps:

- a. loading a HZSM-5 catalyst impregnated with tungsten compound into a fixed bed reactor and flushing the catalyst with nitrogen gas,
- 35 b. heating ethanol liquid at a temperature of 52 -

70°C and a pressure of 1-2 bar to produce ethanol vapor,

c. flowing the heated ethanol vapor with the help of nitrogen carrier gas until it enters the fixed bed reactor that has been loaded with the HZSM-5 catalyst modified with the tungsten-containing compound, and

d. condensing the reaction products to separate ethylene gas from the condensable reaction products through gradual condensation with a temperature range of -5°C to -30°C.

10 In other embodiment of the process of producing ethylene referred to above, the catalytic dehydration reaction of ethanol in said reactor is at a temperature of between 220-300°C and a pressure of 1-2 bar.

15 In other embodiment of the process of producing ethylene referred to above, the process is characterized by being carried out at a total weight hourly space velocity (WHSV) of about 0.3 to about 0.6 per hour of ethanol per weight of catalyst.

20 In further embodiment of the process of producing ethylene referred to above, the process is characterized by being carried out at a total weight hourly space velocity (WHSV) of about 1.3 to about 3.8 per hour of ethanol per weight of catalyst.

25 In other embodiment of the process of producing ethylene referred to above, the HZSM-5 catalyst impregnated with the tungsten-containing compound is a HZSM-5 catalyst impregnated with phosphotungstic acid.

30 In further embodiment of the process of producing ethylene referred to above, the HZSM-5 catalyst impregnated with the tungsten-containing compound is a HZSM-5 catalyst impregnated with phosphotungstic acid having a chemical formula of $H_3PW_{12}O_{40}$.

35 In other embodiment of the process of producing ethylene referred to above, the HZSM-5 catalyst material in the HZSM-5 catalyst impregnated with the tungsten-

containing compound has a Si/Al ratio ranging from 30-62.

In further embodiment of the process of producing ethylene referred to above, the HZSM-5 catalyst material in the HZSM-5 catalyst impregnated with the tungsten-
5 containing compound has a Si/Al ratio ranging from 32-38.

In other embodiment of the process of producing ethylene referred to above, the catalyst material HZSM-5 impregnated with a tungsten-containing compound comprises a tungsten metal content of 0.1 - 2% wt/wt.

10 Other embodiment of the present invention also include any copolymer of ethylene that could produced by any process of producing ethylene referred to above.

Brief Description of the Drawings

15 In order to provide a thorough description of the present invention and to assist in understanding the advantages of the invention, this description is accompanied by the following drawings:

Figure 1 is a flow diagram of the process for producing
20 ethylene through the catalytic dehydration reaction of ethanol using a HZSM-5 catalyst impregnated with tungsten-containing compound according to the present invention.

Figure 2 is a flow diagram showing a method for producing a HZSM-5 catalyst impregnated with tungsten
25 compound according to the present invention.

Figure 3A is a SEM (Scanning Electron Microscopy) image showing the morphology of the HZSM-5 catalyst before being modified with phosphotungstic acid.

Figure 3B shows the results of quantitative analysis
30 using the ZAF Method Standardless Quantitative Analyses on the HZSM-5 catalyst before being modified with phosphotungstic acid.

Figure 4A is a SEM image showing the morphology of the HZSM-5 catalyst after being modified with phosphotungstic
35 acid.

Figure 4B shows the results of quantitative analysis using the ZAF Method Standardless Quantitative Analyses on the HZSM-5 catalyst after being modified with phosphotungstic acid.

5 Figure 5 is a product gas analysis profile curve of the catalytic dehydration reaction of ethanol using HZSM-5 catalyst impregnated with phosphotungstic acid according to the present invention at four different temperatures, namely 220°C, 260°C, 280°C, and 300°C.

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Detailed Description of the Invention

The present invention relates to a process for producing ethylene by dehydration of ethanol using a HZSM-5 catalyst which has been modified with a tungsten-
15 containing compound. In general, the process is illustrated as presented in FIG. 1.

As illustrated diagrammatically in Figure 1, the process of producing ethylene by dehydration of ethanol was carried out according to the present invention, started
20 with the preparation of a reactor into which ethanol would be catalytically dehydrated to ethylene. The reactor used in this process was a fixed bed type reactor, preferably a tubular fixed bed reactor. The reactor preparation was carried out by loading HZSM-5 catalyst impregnated with
25 tungsten-containing compound into the reactor, and then flushing the reactor containing the catalyst to clean the reactor from impurities, such as O₂, CH₄, and CO₂. The flushing was carried out by flowing nitrogen gas through the reactor column at a flow rate of 100 mL/min. Before being
30 fed into the reactor, liquid ethanol was firstly heated outside the reactor at a temperature of around 52 - 70°C and a pressure of 1-2 bar to produce ethanol vapor.

The catalytic dehydration reaction was carried out by flowing the ethanol vapor through a bubbler at a
35 temperature of 62°C using a nitrogen carrier gas with a

flow rate of 50 mL/min. The ethanol vapor was preheated prior to entering the reactor, such that upon reaching the catalyst bed, the reaction temperature was established at one of several set points: 220°C, 240°C, 260°C, 280°C, and 300°C

Prior to detailing the results of the ethylene production process via ethanol dehydration according to the present invention, the catalyst employed in this dehydration will be described. As mentioned above, the catalyst used for the present invention is the HZSM-5 catalyst that has been previously impregnated with a tungsten-containing compound. This catalyst is a catalyst specially made for the process of producing ethylene by dehydration of ethanol according to the present invention. This catalyst was made by the same inventors as the inventors of this present invention and will be applied for a patent application in a separate patent application from this patent application.

The HZSM-5 catalyst being previously impregnated with a tungsten-containing compound is an HZSM-5 catalyst modified with tungsten-containing compounds by wet impregnation of the tungsten-containing compounds into the HZSM-5 catalyst material. Therefore, this catalyst basically consists of a ZSM-5 catalyst (Zeolite Socony Mobile-5) which is so activated that it becomes acidic, namely HZSM-5, and tungsten metal (wolfram) obtained from tungsten compounds. This modified catalyst can be used, among others, for catalytic dehydration reactions of alcohol compounds into ethylene, ether compounds and/or unsaturated hydrocarbons. For the sake of reference, the HZSM-5 catalyst previously impregnated with a tungsten-containing compound referred to hereinafter will also be abbreviated as W/HZSM-5.

Tungsten compounds that can be used to produce the W/HZSM-5 used in the process of producing ethylene

according to the present invention include phosphotungstic acid (phosphorus-tungsten heteropolyacid) (such as $\text{H}_3\text{PW}_{12}\text{O}_{40}$, $\text{H}_6\text{P}_2\text{W}_{21}\text{O}_{71}$, and $\text{H}_6\text{P}_2\text{W}_{18}\text{O}_{62}$), tungstic acid H_2WO_4 , silica-tungsten heteropolyacid, such as $\text{H}_4\text{SiW}_{12}\text{O}_{40}$, and the
5 salt forms of these acids.

One of the preferred compounds used to modify (impregnate) the HZSM-5 catalyst, i.e. to make the W/HZSM-5 catalyst, which can be used in the process of producing ethylene from ethanol according to the present invention
10 is phosphotungstic acid. The selection of phosphotungstic acid as a modifying agent for the HZSM-5 catalyst is based on its excellent ability as a proton donor. Additionally, phosphotungstic acid has good thermal stability that make it suitable for use at high temperatures. In the reaction
15 of ethanol to ethylene, for example, tungsten metal (W) from the tungsten compound acts as a co-catalyst, enhancing the catalytic activity of the aluminum sites already present in HZSM-5. The two metals exhibit analogous functions as Brønsted acid sites, which serve as the active
20 centers for the catalytic reaction. However, W metal may exhibits greater activity than Al metal at low temperatures. The addition of W as an acid site enables the catalytic reaction to occur at a lower temperature compared to that utilizing HZSM-5 alone.

25 Among the above mentioned phosphotungstic acids, the preferred one for use in the preparation of tungsten-containing compound-embedded HZSM-5 catalyst (W/HZSM-5), which is then used in the ethylene production process according to the present invention, is phosphotungstic acid
30 with the chemical formula $\text{H}_3\text{PW}_{12}\text{O}_{40}$. In this invention, a W/HZSM-5 catalyst was prepared by wet impregnation of phosphotungstic acid $\text{H}_3\text{PW}_{12}\text{O}_{40}$ solution on HZSM-5 particles. The HZSM-5 catalyst already impregnated with the phosphotungstic acid was then calcined at 350°C to remove
35 the solvent used and to activate its catalytic sites. The

resulting HZSM-5 catalyst so modified with phosphotungstic acid (W/HZSM-5) was able to attain 0.1% to 2%, preferably 1%, tungsten impregnation on HZSM-5. This modified catalyst showed high catalyst activity in the temperature range of 220-300°C and was able to achieve ethanol to ethylene conversion of more than 95% and selectivity of more than 94%.

The method of making HZSM-5 catalyst containing tungsten compounds (W/HZSM-5) used in the process of making ethylene according to the invention will be described in more detail with reference to the flow chart as shown in Figure 2.

As shown in Figure 2, the method of preparing W/HZSM-5 catalyst begins with a pre-treatment step (2) of the HZSM-5 catalyst raw material used as a support for the material to be impregnated (i.e. tungsten from tungsten compounds). The HZSM-5 material used in this invention has a Si/Al ratio ranging from 30-62, preferably 32-38. At this step, the HZSM-5 material, initially in pellet form, is subjected to a size reduction process until uniform pellets with a length of approximately 5-10 mm are obtained. Afterwards, the HZSM-5 material that has been cut into pieces is heated at a temperature of 50-60°C and a vacuum pressure of -0.5 bar. This heat and pressure treatment is taking place for about 15 minutes. Meanwhile, the preparation step of the impregnating material (4) is also carried out. In this preparation step (4), the tungsten compound is dissolved in distilled water with a predetermined composition so that the ratio of tungsten metal contained in the HZSM-5 material is 0.1% to 2% weight/weight, 0.9 to 1.1% weight/weight, preferably 1% weight/weight. In the example embodiment of the invention shown in Figure 2, the tungsten compound used is phosphotungstic acid ($\text{H}_3\text{PW}_{12}\text{O}_{40}$).

The impregnation step (6) is carried out after the

pre-treatment step of HZSM-5 material (2) and the preparation step of impregnating material (4) being completed. The impregnation is conducted by dripping phosphotungstic acid solution into the HZSM-5 material at a temperature of 50°C and atmospheric pressure. Subsequently, the HZSM-5 material that has been completely wetted is left standing at a temperature of 50°C. The impregnated material is then kept at room temperature and atmospheric pressure for 24 hours.

After the impregnation step (6), the next step of the W/HZSM-5 catalyst preparation method is the drying step (8). In the drying step (8), the HZSM-5 material that has been impregnated with phosphotungstic acid is completely dried in a drying oven at a temperature of 60-70°C for 24 hours. The next step is the calcination step (10). In this calcination step (10), the HZSM-5 material that has been impregnated with phosphotungstic acid and dried is heated at a heating rate of 2°C/min until it reaches a temperature of 350°C. The material is then maintained at a temperature of 350°C.

The HZSM-5 catalyst plays a role in protonating the hydroxyl group (OH)- which then being converted into water molecules (H₂O). The conjugate base of the catalyst then releases protons (H⁺) from the methyl group to form ethylene. The presence of tungsten metal (W) from phosphotungstic acid accelerates this process.

A phosphotungstic-acid-impregnated HZSM-5 catalyst (W/HZSM-5) was obtained by the preparation method as mentioned above, and surface characterization, pore structure analysis, and elemental distribution tests were conducted on the catalyst using SEM (Scanning Electron Microscopy) and EDX (Energy-Dispersive X-ray Spectroscopy) instruments. The SEM instrument depicted the sample surface through scanning using high-energy electron beams in a raster scan pattern. Figure 3A shows the SEM image

with a magnification of 10000x, and Figure 3B shows the results of the analysis using the EDX tool for the HZSM-5 material before being modified with phosphotungstic acid. Based on the results of the EDX analysis, it can be seen that HZSM-5 contains the elements C (carbon), O (oxygen), Si (silica), and Al (alumina), and the comparison of the mass percentage of the constituent elements: C = 8.38%; O = 47.63%; Si = 24.89%; and Al = 19.10%.

Figure 4A shows the SEM image with 10000x magnification and EDX analysis of W/HZSM-5 material, namely HZSM-5 impregnated with phosphotungstic acid prepared by the method referred to above. Based on the EDX analysis shown in Figure 4B, it can be seen that W/HZSM-5 contains the elements C (carbon), O (oxygen), Si (silica), Al (alumina), and W (tungsten). The mass percentage comparison of the constituent elements is as follows: C = 6.92%; O = 38.74%; Si = 23.94%; Al = 17.66%; and W = 12.74%. Furthermore, the mole percentage of the constituent elements is as follows: C = 12.60%; O = 52.94%; Si = 18.64%; Al = 14.31%; and W = 1.51%.

Several tests have been conducted to determine the performance of the ethylene production process by catalytic dehydration reaction of ethanol using an HZSM-5 catalyst impregnated with tungsten-containing compound (W/HZSM-5) according to the present invention. The ethylene production process was carried out according to the process steps as mentioned above. The catalytic dehydration reaction of ethanol in the reactor occurred at a temperature ranging from 220-300°C and a pressure of 1-2 bar. The measurements of ethanol conversion and ethylene selectivity were taken at several reaction temperatures. Some tests were carried out using HZSM-5 catalyst impregnated with tungsten-containing compound (W/HZSM-5) and non-impregnated HZSM-5 catalyst as a comparison. The material of HZSM-5 catalyst impregnated with tungsten-containing

compound, W/HZSM-5, had a tungsten metal content of 0.1-2% wt/wt, preferably 1% wt/wt. The reactor used in the process was a tubular fixed bed reactor. The amount of the W/HZSM-5 catalyst and the non-impregnated HZSM-5 catalyst used in this test were approximately 2.2 g each. The total weight flow rate of ethanol per weight of catalyst (weight hourly space velocity; WHSV) in this dehydration reaction test was approximately 0.3 to 0.6 per hour. The total weight flow rate per weight of catalyst (weight hourly space velocity; WHSV) in the process was approximately 1.3 to 3.8 per hour.

For the purpose of this test, the product gas of the catalytic dehydration reaction of ethanol was flowed through an impinger containing water to absorb the remaining ethanol vapor that does not react. Afterward, the product gas was flowed through a condenser to gradually separate (condensate) the ethylene gas from other product gases (by-products) that was non-condensable. This sequential condensation was carried out with a temperature range of -5°C to -30°C. The quantity of the ethylene gas produced was then analyzed using a Shimadzu GC-8A Gas Chromatography (TCD Detector Type), and the analysis of the remaining ethanol from the solution in the impinger was carried out using a Shimadzu GC-14B Gas Chromatography (FID Detector Type).

The test results of the ethylene production process through the catalytic dehydration reaction of ethanol using the W/HZSM-5 catalyst and the non-impregnated HZSM-5 catalyst at various reaction temperatures are shown in Table 1. From Table 1 it can be seen that for various fairly low reaction temperatures, namely 220-300°C, the process using the HZSM-5 catalyst impregnated with a tungsten-containing compound (W/HZSM-5) was able to produce a fairly high ethanol conversion and ethylene selectivity when compared to a similar process using the

HZSM-5 alone (i.e. without impregnation). The test results also show that the reaction process of the ethylene production through the catalytic dehydration reaction of ethanol using the HZSM-5 catalyst that were impregnated with a tungsten-containing compound (W/HZSM-5) was able to produce ethanol conversion above 96% with ethylene selectivity above 94% for a relatively low reaction temperature, namely 220-300°C.

The profile analysis curve of gas produced in the catalytic dehydration reaction of ethanol using the W/HZSM-5 catalyst at (a) reaction temperature of 220°C, (b) reaction temperature of 260°C, (c) reaction temperature of 280°C, and (d) reaction temperature of 300°C are shown in Figure 5.

It should be noted that the above description is merely intended to describe how the process of producing ethylene through the catalytic dehydration reaction of ethanol using the W/HZSM-5 catalyst, i.e. HZSM-5 catalyst impregnated with a tungsten-containing compound, according to the present invention. After reading the description, it is expected that a person skilled in the art will of course know that the process of producing ethylene mentioned above may be modified or varied without going beyond the scope of the present invention. Therefore, the scope of the present invention is not limited to the above description, but to the claims shown below.

Table 1

Material	Ethanol Conversion (%)					Ethylene Selectivity (%)				
Temperature	220°C	240°C	260°C	280°C	300°C	220°C	240°C	260°C	280°C	300°C
HZSM-5	51.26	74.33	69.57	69.60	93.82	0.00	17.53	30.86	55.76	92.24
1%-W/HZSM-5	96.20	97.77	99.93	99.95	99.93	94.20	95.55	99.84	99.90	99.88

*Overall WHSV: 1.8/hour; Ethanol WHSV: 0.3/hour

Claims

1. A process of producing ethylene through a catalytic dehydration reaction of ethanol with the following steps:

- a. loading a HZSM-5 catalyst impregnated with tungsten compound into a fixed bed reactor and flushing the catalyst with nitrogen gas,
- b. heating ethanol liquid at a temperature of 52 - 70°C and a pressure of 1-2 bar to produce ethanol vapor,
- c. flowing the heated ethanol vapor using nitrogen as a carrier gas until it enters the fixed bed reactor that has been loaded with the HZSM-5 catalyst modified with the tungsten-containing compound, and
- d. condensing the reaction products to separate ethylene gas from the condensable reaction products through gradual condensation with a temperature range of -5°C to -30°C.

2. The process according to claim 1, wherein the catalytic dehydration reaction of ethanol in said reactor is at a temperature of between 220-300°C and a pressure of 1-2 bar.

3. The process according to claim 1 or 2 wherein the total weight hourly space velocity (WHSV) of ethanol per weight of catalyst in the process is about 0.3 to about 0.6 per hour.

4. The process according to claim 1 or 2 wherein the total weight hourly space velocity (WHSV) in said process is about 1.3 to about 3.8 per hour.

5. The process according to previous claims wherein the HZSM-5 catalyst modified with the tungsten-containing compound is a HZSM-5 catalyst impregnated with by phosphotungstic acid.

6. The process according to claim 5 wherein the HZSM-5 catalyst modified with the tungsten-containing compound is a catalyst HZSM-5 impregnated with phosphotungstic acid

having a chemical formula of $\text{H}_3\text{PW}_{12}\text{O}_{40}$.

7. The process according to claim 5 and 6 wherein the catalyst material HZSM-5 in the HZSM-5 catalyst is impregnated with a tungsten-containing compound having a
5 Si/Al ratio ranging from 30-62.

8. The process according to claim 7 wherein the catalyst material HZSM-5 in the HZSM-5 catalyst is impregnated with a tungsten-containing compound having a Si/Al ratio of 32-38.

10 9. The process according to claim 5, 6, 7 and 8 wherein the catalyst material HZSM-5 impregnated with a tungsten-containing compound comprises a tungsten metal content of 0.1 - 2% wt/wt.

15 10. A copolymer of ethylene obtainable by the process of any one of the previous claims.