

PATENT ASSIGNMENT COVER SHEET

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SUBMISSION TYPE:	NEW ASSIGNMENT
NATURE OF CONVEYANCE:	ASSIGNMENT
CONVEYING PARTY DATA	
Name	Execution Date
STEVEN L. SUIB	12/10/2015
D. A. SAMINDA DHARMARATHNA	12/11/2015
LAKSHITHA R. PAHALAGEDARA	12/10/2015
RECEIVING PARTY DATA	
Name:	UNIVERSITY OF CONNECTICUT
Street Address:	400 FARMINGTON AVENUE, MC-6400
Internal Address:	OFFICE OF THE VICE PRESIDENT FOR RESEARCH
City:	FARMINGTON
State/Country:	CONNECTICUT
Postal Code:	06032
PROPERTY NUMBERS Total: 1	
Property Type	Number
Application Number:	14971404
CORRESPONDENCE DATA	
Fax Number:	(203)327-6401
<i>Correspondence will be sent to the e-mail address first; if that is unsuccessful, it will be sent using a fax number, if provided; if that is unsuccessful, it will be sent via US Mail.</i>	
Phone:	203 327 4500
Email:	docketing@OGRP.com
Correspondent Name:	OHLANDT, GREELEY, RUGGIERO & PERLE, LLP
Address Line 1:	ONE LANDMARK SQUARE, 10TH FLOOR
Address Line 4:	STAMFORD, CONNECTICUT 06901
ATTORNEY DOCKET NUMBER:	0010289USU/2480
NAME OF SUBMITTER:	CHARLES N.J. RUGGIERO
SIGNATURE:	/Charles N.J. Ruggiero/
DATE SIGNED:	12/18/2015
This document serves as an Oath/Declaration (37 CFR 1.63).	
Total Attachments: 37	
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OHLANDT, GREELEY, RUGGIERO & PERLE, L.L.P.

ONE LANDMARK SQUARE, 10TH FLOOR
STAMFORD, CONNECTICUT 06901-2682

Telephone (203) 327-4500
www.ogrp.com

**COMBINED DECLARATION AND ASSIGNMENT UNDER 37 C.F.R. §1.63 FOR
UTILITY OR DESIGN PATENT APPLICATION USING AN APPLICATION DATA SHEET**

OGRP Rev. No. A1A/01a

This Declaration and Assignment, when present, are directed to:

Title of Invention: METHOD FOR REMOVING SOOT FROM EXHAUST GASES

Attorney Docket No.: 0010289USU/2480

- ☒ The specification of which is attached hereto, or
- ☐ United States application or PCT international application number
_____ filed on _____.

I hereby authorize and request the delegated attorneys or agents or those of the Assignee, if any, to insert above the application number and filing date of the above-identified application when known.

Declaration: In accordance with the requirements under 35 U.S.C §115, as a below named inventor, I hereby declare that:

- The above-identified application was made or authorized to be made by me.
- I have reviewed and understand the contents of the above-identified application, including the claims, as required by 37 C.F.R. §1.63.
- I believe that I am the original inventor when only one inventor is named below or an original joint inventor when more than one inventor is named below of a claimed invention in the above-identified application.
- I am aware of the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to the patentability of the above-identified application as defined in 37 C.F.R. §1.56.
- I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. §1001 by fine or imprisonment of not more than five (5) years, or both.

COMBINED DECLARATION AND ASSIGNMENT UNDER 37 C.F.R. §1.63 (CONTINUED)

Assignment:

- ☐ The above-identified application is not subject to an assignment, or
- ☑ The above-identified application is subject to the following assignment.

Whereas, UNIVERSITY OF CONNECTICUT, a corporation of a public institution of higher education having a place of business at Office of the Vice President for Research, 400 Farmington Avenue, MC-6400, Farmington, Connecticut 06032 (the "Assignee"), desires to acquire, and each undersigned inventor desires to grant and hereby grants to the Assignee, the entire worldwide right, title, and interest in and to the above-identified application and in and to any and all patent applications and patents directed thereto.

Now, therefore, for good and valuable consideration, the receipt and sufficiency thereof being hereby acknowledged, each undersigned inventor hereby sells or has sold, assigns or has assigned, and otherwise transfers or has transferred to the Assignee, its successors, legal representatives, and assigns, the entire worldwide right, title, and interest in and to the Invention, the above-identified United States patent application, and any and all other patent applications and patents for the above-identified application that may be applied for or granted therefore in the United States and in all foreign countries and jurisdictions, including all divisions, continuations, reissues, reexaminations, renewals, extensions, counterparts, substitutes, and extensions thereof, and all rights of priority resulting from the filing of such applications and granting of such patents.

In addition, each undersigned inventor hereby authorizes and requests the Director of the United States Patent and Trademark Office to issue any United States Patent, and foreign patent authorities to issue any foreign patent, granted for the Invention, to the Assignee, its successors, legal representatives, and assigns, the entire worldwide right, title, and interest in and to the same to be held and enjoyed by the Assignee, its successors, legal representatives, and assigns to the full end of the terms for which any and all such patents may be granted, as fully and entirely as would have been held and enjoyed by the undersigned had this Assignment not been made; and each undersigned inventor agrees to execute any and all documents and instruments and perform all lawful acts reasonably related to recording this Assignment or perfecting title to the above-identified application and all related patents and applications, in the Assignee, its successors, legal representatives, and assigns, whenever requested by the Assignee, its successors, legal representatives, or assigns.

Each undersigned inventor acknowledges his or her obligations to sell, assign, and transfer the rights under this Assignment to the Assignee and is unaware of any reason why he or she may not have the full and unencumbered right to sell, assign, and transfer the rights hereby sold, assigned, and transferred, and has not executed, and will not execute, any document or instrument in conflict herewith.

COMBINED DECLARATION AND ASSIGNMENT UNDER 37 C.F.R. §1.63 (CONTINUED)

Add Inventor

Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	Steven	L.	SUIB	
Signature: <u>Steven L. Suib</u>				
Date: <u>12/10/15</u>				

Remove Inventor

Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	D. A. Saminda		DHARMARATHNA	
Signature: _____				
Date: _____				

Remove Inventor

Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	Lakshitha	R.	PAHALAGEDARA	
Signature: _____				
Date: _____				

Remove Inventor

OHLANDT, GREELEY, RUGGIERO & PERLE, L.L.P.

ONE LANDMARK SQUARE, 10TH FLOOR
STAMFORD, CONNECTICUT 06901-2682

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Title of Invention: METHOD FOR REMOVING SOOT FROM EXHAUST GASES

Attorney Docket No.: 0010289USU/2480

- ☒ The specification of which is attached hereto, or
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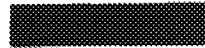
Whereas, UNIVERSITY OF CONNECTICUT, a corporation of a public institution of higher education having a place of business at Office of the Vice President for Research, 400 Farmington Avenue, MC-6400, Farmington, Connecticut 06032 (the "Assignee"), desires to acquire, and each undersigned inventor desires to grant and hereby grants to the Assignee, the entire worldwide right, title, and interest in and to the above-identified application and in and to any and all patent applications and patents directed thereto.

Now, therefore, for good and valuable consideration, the receipt and sufficiency thereof being hereby acknowledged, each undersigned inventor hereby sells or has sold, assigns or has assigned, and otherwise transfers or has transferred to the Assignee, its successors, legal representatives, and assigns, the entire worldwide right, title, and interest in and to the Invention, the above-identified United States patent application, and any and all other patent applications and patents for the above-identified application that may be applied for or granted therefore in the United States and in all foreign countries and jurisdictions, including all divisions, continuations, reissues, reexaminations, renewals, extensions, counterparts, substitutes, and extensions thereof, and all rights of priority resulting from the filing of such applications and granting of such patents.

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COMBINED DECLARATION AND ASSIGNMENT UNDER 37 C.F.R. §1.63 (CONTINUED)



Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	Steven	L.	SUIB	
Signature: _____				
Date: _____				



Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	D. A. Saminda		DHARMARATHNA	
Signature: _____				
Date: 12/11/15				



Inventor Information :				
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Signature: _____				
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OHLANDT, GREELEY, RUGGIERO & PERLE, L.L.P.

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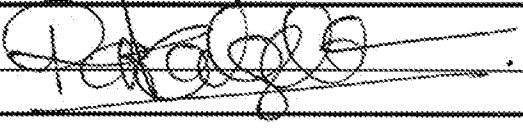
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	Steven	L.	SUIB	
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Remove Inventor

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Date: _____				

Remove Inventor

Inventor Information :				
Prefix	Given Name	Middle Name	Family Name	Suffix
	Lakshitha	R.	PAHALAGEDARA	
Signature: 				
Date: 12.10.15				

Remove Inventor

METHOD FOR REMOVING SOOT
FROM EXHAUST GASES

RELATED APPLICATION

[0001] This application claims the benefit of U.S. Provisional Application Serial No. 62/093,180, filed on December 17, 2014, which is incorporated herein by reference in its entirety.

GOVERNMENT LICENSE RIGHTS

[0002] This invention was made with government support under contract 5226640 awarded by U.S. Department of Energy. The government has certain rights in the invention.

BACKGROUND

1. Field of the Disclosure

[0003] This disclosure relates to a method for removing soot from exhaust gases. In particular, this disclosure relates to a method for oxidizing soot from diesel exhaust gas from a diesel engine, a diesel exhaust gas treatment system, a method of providing a catalyst composition on a diesel particulate filter, and a catalyst composition for facilitating soot oxidation. The catalyst composition is a doped or undoped manganese oxide octahedral molecular sieve (OMS-2) material (e.g., cobalt doped manganese oxide octahedral molecular sieve (OMS-2) material).

2. Discussion of the Background Art

[0004] In general, diesel engines are advantageous because they have various uses due to their high power and ability to be operated even under high loads. However, since exhaust gases discharged from such diesel engines are a major source of air pollution, their allowable discharge standard becomes more and more strict throughout the world. The main pollutants from diesel engines include

fine soot particulates, hydrocarbons, carbon monoxide, soluble organic solvents, and nitrogen oxides. In particular, the soot particulates directly impact life, such as increasing a generation rate of cancer of the respiratory organs.

[0005] In recent years, environmental regulations in the United States and Europe restricting diesel particulate emissions have necessitated improvements in the removal of particulates from diesel engine emissions. Such particulates generally consist of carbonaceous particulates in the form of soot.

[0006] At present, there are two major diesel particulate filter (DPF) regeneration techniques; active regeneration and passive regeneration. In active DPF regeneration the particulate matter is periodically oxidized either using a heater/a flame based burner at elevated temperatures (above 600°C) or by increasing the exhaust gas temperature. This flame based technique possesses serious fire hazard and also due to the elevated temperature, the filter matrix could breakdown and cause damage to the internal engine parts. Another drawback of this method is the poor regeneration efficiency, resulting in deposition of up to 35% of soot on the filter. This method also consumes a large amount of energy during the heating process thereby decreasing the fuel efficiency of the engine.

[0007] In passive regeneration, the particulate matter is oxidized by means of an on-going catalytic oxidation process. The method is both economically and environmentally preferred since it requires no additional energy source, occurs at exhaust gas temperature, very simple, effective, and fuel efficient. However, finding a catalyst which is capable of decreasing the soot combustion temperature from 600°C to diesel engine exhaust temperature (typically 150 - 400°C) and also carryout complete oxidation of soot to CO₂ without toxic CO gas is challenging.

[0008] In most cases, the filter is coated with precious metals including platinum group metals (PGM) such as Pt and Pd. However, these metals are very expensive and they also highly active in undesirable reactions such as oxidation of

SO₂ to SO₃. In addition to PGM, Ag with an active metal (Cu, La, Co, Ni, Mn, Fe, Ce) oxide component has also been used in DPF coatings. However, these materials are capable of oxidizing soot at temperatures between 500 – 600°C, which are much higher than the diesel engine exhaust temperatures. Therefore, additional energy is required to drive the oxidation.

[0009] Furthermore, currently most of the catalysts generate a considerable amount of CO during the combustion of diesel soot which, is a byproduct from incomplete oxidation and possesses serious health hazard. In addition, most of the currently used catalysts require a high catalyst to soot ratio which reduces its efficiency in the real applications. Thus, it is desirable to develop a diesel soot oxidation catalyst which is highly active at diesel engine exhaust temperatures, highly selective towards CO₂, much greener, highly efficient, and less expensive.

[0010] The present disclosure provides many advantages over the prior art, which shall become apparent as described below.

SUMMARY OF THE DISCLOSURE

[0011] This disclosure relates in part to a method for oxidizing soot from diesel exhaust gas from a diesel engine. The method comprises: providing a diesel particulate filter for receiving the diesel exhaust gas; coating a catalyst composition on the diesel particulate filter; wherein the catalyst composition comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2); and contacting the soot from the diesel exhaust gas with the catalyst coated diesel particulate filter at a temperature sufficient to oxidize the soot to carbon dioxide.

[0012] This disclosure also relates in part to a diesel exhaust gas treatment system. The diesel exhaust gas treatment system comprises: a diesel particulate filter for receiving diesel exhaust gas from a diesel engine and collecting soot; and a catalyst composition coated on the diesel particulate filter; wherein the catalyst

composition comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

[0013] This disclosure further relates in part to a method of providing a catalyst composition on a diesel particulate filter. The method comprises: providing a diesel particulate filter; and applying a catalyst composition for facilitating soot oxidation on the diesel particulate filter; wherein the catalyst comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

[0014] This disclosure yet further relates in part a catalyst composition for facilitating soot oxidation. The catalyst comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

[0015] Further objects, features and advantages of the present disclosure will be understood by reference to the following drawings and detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] FIG. 1 graphically shows the effect of temperature on the soot oxidation activity for noncatalytic (NON), Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM) in accordance with Example 4.

[0017] FIG. 2 shows the oxidation temperature of soot with noncatalytic (NON), Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM) in accordance with Example 4.

[0018] FIG. 3 graphically shows activation energies for non-catalyzed (NON), and catalyzed [Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized

undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM)] soot oxidation reactions in accordance with Example 4.

[0019] FIG. 4 graphically shows selectivity towards carbon dioxide as a function of temperature for non-catalyzed (NON) and Co-doped cryptomelane catalyzed (Co-OMS-2) reactions in accordance with Example 4.

[0020] FIG. 5 shows XRD patterns for the reaction mixture consists of Co- doped cryptomelane (Co-OMS-2), soot, and silicon carbide, at different reaction temperatures in accordance with Example 4.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0021] All numerical values within the detailed description and the claims herein are modified by "about" or "approximately" the indicated value, and take into account experimental error and variations that would be expected by a person having ordinary skill in the art.

[0022] This disclosure relates to a catalyst for selective oxidation of diesel particulate matter (soot) to carbon dioxide, which is to be used in the catalytic coating for DPFs. The catalyst is 100% selective to carbon dioxide and no carbon monoxide is obtained as an oxidation product. In addition, the catalyst prepared according to the present disclosure assists the oxidation reaction to achieve 100% conversion at a lower temperature (~350°C) using a lower catalyst to soot weight ratio (2:1) as compared to previously reported catalysts. The catalyst preparation and the product thereof are essentially free of rare earth metals, precious metals, highly toxic metals, highly acidic/basic conditions.

[0023] The catalyst of this disclosure can be used in the after treatment process (carbon combustion) of the diesel engine exhaust system by the automotive industry. The catalyst can be used as a coating in DPFs to oxidize the undesirable soot deposits and regenerate the filter.

[0024] The present disclosure does not require high temperatures and pressures and operates under diesel engine exhaust temperatures. Thus, the use of an external heater is not required. Therefore, the periodic regeneration of the DPF or an extra attention is not needed. This increases the efficiency of the diesel engine and reduces the wear of the diesel particulate filter. Also, no additional fuel is needed for regeneration process. This greatly reduces the cost for the regeneration process. Furthermore, since the starting materials of the new catalyst are cheap, the cost for the catalyst coated DPF could be reduced to a great extent.

[0025] The catalyst of this disclosure is highly active at diesel engine exhaust temperatures, environmentally benign, and does not include noble metals, hence less expensive. The catalytic process is 100% selective towards CO₂. The reaction is truly catalytic. Moreover, the catalyst is stable at high temperatures. Also, the catalyst is highly active even at ambient pressure and is independent of the flow rate, hence, the catalytic process is predictable. Thus, the disclosure is potentially attractive for large scale industrial applications.

[0026] The OMS-2 catalyst material of this disclosure can burn off the soot particulates accumulated on a DPF at the temperature of diesel exhaust (low temperature ~350°C). The oxidation produces 100% CO₂ rather than CO as the oxidation product. Cobalt doped cryptomelane (Co-OMS-2) further reduces the operating temperature. This disclosure has application on the exhaust system of diesel vehicles or other diesel burning equipment (generators).

[0027] DPFs capture particle emissions through a combination of filtration mechanisms, such as diffusional deposition, inertial deposition, or flow-line interception. Collected particulates are removed from the filter, continuously or periodically, through thermal regeneration. Diesel filters are highly effective in controlling solid particulate emissions—including solid particle numbers—but may be ineffective in controlling liquid fractions of PM emission. Filters were

first commercialized as retrofit devices, followed by a wide scale adoption on new light-duty and heavy-duty diesel engines in both highway and non-road applications.

[0028] The catalyst of this disclosure does not require noble or rare earth metals and performs at low temperature. Therefore an external heater on the particulate filter is not required for the vehicle exhaust system.

[0029] The manganese oxide is preferably a manganese oxide octahedral molecular sieve (OMS) material. The cobalt doped manganese oxide octahedral molecular sieve (OMS) preferably has a Cryptomelane type structure.

[0030] The cobalt in the cobalt doped manganese oxide octahedral molecular sieve (OMS) material is present in an amount of from about 0.1 weight percent to about 25 weight percent

[0031] The metal oxide catalyst material useful in the process of this disclosure catalyzes the oxidation of soot to carbon dioxide. The presence of the metal oxide material ensures that the product begins to work immediately after being placed in contact with diesel exhaust gas. The metal oxide can be a manganese oxide, manganese hydroxide, iron oxide, iron hydroxide, zinc oxide, zinc hydroxide, and combinations thereof. The use of manganese oxide octahedral molecular sieve (OMS) material is preferred. The use of a doped (e.g., cobalt) manganese oxide octahedral molecular sieve (OMS) material is more preferred.

[0032] The concentration of the manganese oxide octahedral molecular sieve (OMS) catalyst material used in the process of this disclosure can vary over a wide range and need only be at a concentration sufficient to form the oxidation catalyst composition. The manganese oxide octahedral molecular sieve (OMS) material can be present in a molar concentration ratio of from about 1 M to about

10 M, preferably from about 1 M to about 5 M, and more preferably from about 0.5 M to about 1 M (based on a total volume).

[0033] One or more metal dopant precursors are useful in the processes of this disclosure. Physicochemical properties of the manganese oxide octahedral molecular sieve (OMS) catalyst materials of this disclosure can be controlled by the use of metal dopant precursors in the synthesis thereof. The metal dopant precursors useful in this disclosure makes the tuning of physicochemical properties of the manganese oxide octahedral molecular sieve (OMS) catalyst materials possible by controlling, for example, surface area, pore size and pore volume.

[0034] Illustrative metal dopant precursors include, for example, metal halides, metal phosphates, metal acetates, metal nitrates, metal alkoxides, and M_xO_y oxides that are capable of dissolving in HNO_3 . More particularly, metal precursors include, for example, metal halides such as $M(NO_3)_x \cdot yH_2O$ ($M = Mn, Fe, Co, Cu, Zn, Si$), $HfCl_4$ or $NbCl_4$, tetraethyl orthosilicate (TEOS), tetramethyl orthosilicate (TMOS), titanium isopropoxide, zirconium n-propoxide, zirconium butoxide, $Ce(NO_3)_3 \cdot 6H_2O$, $Gd(NO_3)_3 \cdot 6H_2O$, $Sm(NO_3)_3 \cdot 6H_2O$, and the like.

[0035] The metal dopant precursors useful in the process of this disclosure include precursors of transition metals, Lanthanide metals, post-transition metals, metalloids, and mixtures thereof. For example, the transition metal precursors comprise Group 3-12 transition metal precursors, in particular, Sc, Y, La, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Mn, Tc, Re, Fe, Ru, Os, Co, Rh, Ir, Ni, Pd, Pt, Cu, Ag, Au, Zn, Cd and Hg precursors. In an embodiment, the transition metal precursors are selected from Group 6-12 transition metal precursors including Cr, Mo, W, Mn, Tc, Re, Fe, Ru, Os, Co, Rh, Ir, Ni, Pd, Pt, Cu, Ag, Au, Zn, Cd and Hg precursors. Preferably, the Group 6-12 transition metal precursors include Mn, Fe, Co, Ni, Cu and Zn precursors. The Lanthanide metal precursors include La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu precursors, or any

mixture thereof. The post-transition metal precursors include Al, Ga, In, Tl, Sn, Pb and Bi precursors, or any mixture thereof. The metalloid precursors include B, Si, Ge, As, Sb, Te, Po and At precursors, or any mixture thereof.

[0036] The concentration of the metal dopant precursors used in the process of this disclosure can vary over a wide range and need only be at a concentration sufficient to form the doped manganese oxide octahedral molecular sieve (OMS) materials. The metal precursors can be present in a molar concentration ratio of from about 1×10^{-2} M to about 10 M, preferably from about 1×10^{-1} M to about 5 M, and more preferably from about 5×10^{-1} M to about 1 M (based on a total volume).

[0037] Preferred metal dopant precursors include $M(\text{NO}_3)_x$ ($M = \text{Co}$).

[0038] The catalyst composition to soot weight ratio is from about 1.5:1 to about 3:1, preferably about 2:1.

[0039] The process conditions for doping the manganese oxide octahedral molecular sieve (OMS) catalyst materials with the metal dopant precursor, such as temperature, pressure and contact time, may vary greatly and any suitable combination of such conditions may be employed herein. In a preferred embodiment, the process conditions are sufficient to tune the structural properties of the doped manganese oxide octahedral molecular sieve (OMS) catalyst materials. The doping temperature may be between about -80°C to about 150°C , and most preferably between about 20°C to about 80°C . Normally the doping is carried out under ambient pressure and the contact time may vary from a matter of seconds or minutes to a few hours or greater. The reactants can be added to the reaction mixture or combined in any order. The stir time employed can range from about 0.1 to about 400 hours, preferably from about 1 to 75 hours, and more preferably from about 4 to 16 hours.

[0040] The method of making the doped manganese oxide octahedral molecular sieve (OMS) catalyst materials involves mixing all the constituents in ambient temperature to achieve a homogenous mix. The amount of each constituent can be varied within the ranges disclosed above. Any of a variety of devices can be used to stir or mix the products, including paddle mixers, tumblers, and a variety of other stirrers or mixers.

[0041] In addition, the oxidation catalyst of the present disclosure may be used by directly applying it to an exhaust pipe for the discharge of exhaust gases, or by applying it to a wall of a device for filtering soot particulates or a honeycombed dust filter module for purifying exhaust gases. Thereby, the fine soot particulates adhere on the surface of the oxidation catalyst thus applied, and thus, they are oxidized by oxygen present in the exhaust gases and removed. This procedure may be explained as follows:



[0042] In addition, the present disclosure provides a method of removing fine soot particulates, which comprises contacting fine soot particulates of exhaust gases with the oxidation catalyst to oxidize the fine soot particulates using oxygen in exhaust gases at 150-550°C, preferably 150-450°C, and more preferably 150-350°C, thus removing fine soot particulates.

[0043] The oxidation catalyst of the present disclosure can be directly applied to an exhaust pipe for discharging exhaust gases, or is applied to a wall of a device for filtering soot particulates or a honeycombed module for purifying exhaust gases. Thereby, the oxidation catalyst contacts fine soot particulates of exhaust gases, and thus, the soot particulates are oxidized using oxygen at 150°C or higher, thus removing fine soot particulates. Meanwhile, the fine particulates, precipitated due to the incomplete combustion at a temperature lower than 150°C, begin to burn in the presence of the catalyst of the present disclosure as the

temperature of the diesel engine is increased to 150°C or higher. Further, the soot particulates are combusted without catalysts at a temperature of higher than 650°C.

[0044] The oxidation catalyst of the present disclosure may be used to remove fine soot particulates from exhaust gases of diesel engines, incinerators, or boilers, at low temperatures. Further, since the above catalyst can exhibit thermal durability, its activity may be stably maintained even under thermal stress for a long time period. Also, the catalyst eliminates the danger of poisoning from sulfur compounds present in exhaust gases, and can stably maintain its activity.

[0045] The catalyst composition of the present disclosure provides comparable or better soot oxidation properties than commercial formulations having noble metal loadings. In addition, when coated onto a diesel particulate filter, the catalyst composition provides good thermal stability, minimal backpressure, and high surface area. By "thermal stability" it is meant that the catalyst maintains its activity or oxidation performance even after exposure to high temperatures. By "backpressure," it is meant the resistance to flow which is caused when the filter pores become blocked or are decreased in size, e.g., from the accumulation of soot or the coating of the catalyst onto the filter. By "minimal backpressure" it is meant that, when coated onto the filter, the catalyst coating of the present disclosure increases the backpressure of the filter only slightly.

[0046] The doped or undoped manganese oxide octahedral molecular sieve (OMS-2) catalyst of this disclosure can be regenerated at a temperature and for a period to time sufficient to substantially oxidize the soot to carbon dioxide. In an embodiment, the regenerating can be conducted at a temperature of about 350°C or less. In another embodiment, the regenerating can be conducted at a temperature of the diesel exhaust gas.

[0047] In the above detailed description, the specific embodiments of this disclosure have been described in connection with its preferred embodiments. However, to the extent that the above description is specific to a particular embodiment or a particular use of this disclosure, this is intended to be illustrative only and merely provides a concise description of the exemplary embodiments. Accordingly, the disclosure is not limited to the specific embodiments described above, but rather, the disclosure includes all alternatives, modifications, and equivalents falling within the true scope of the appended claims. Various modifications and variations of this disclosure will be obvious to a worker skilled in the art and it is to be understood that such modifications and variations are to be included within the purview of this application and the spirit and scope of the claims.

[0048] All reactions in the following examples were performed using as-received starting materials without any purification.

Synthesis of Cryptomelane (K-OMS-2) and Co Doped K-OMS-2

Example 1

Hydrothermal Synthesis of K-OMS-2

[0049] Potassium sulfate, potassium persulfate, and manganese sulfate monohydrate in the molar ratio of 3:3:2 (totaling 32 mmol) were dissolved in 70 mL of DDW. The reaction mixture was then transferred into a 125 mL hydrothermal reactor vessel and kept in an oven at 240 °C for 4 days. Finally, the dark brown slurry was filtered and washed with DDW until the filtrate was neutral. The solid catalyst was dried at 120 °C overnight before use.

Hydrothermal Synthesis of Cobalt Doped K-OMS-2

[0050] Potassium sulfate, potassium persulfate, and manganese sulfate monohydrate in the molar ratio of 3:3:2 (totaling 32 mmol) were dissolved in 70 mL of DDW. Then 2 mmol of cobalt nitrate was added to the reaction mixture. The reaction mixture was then transferred into a 125 mL hydrothermal reactor vessel and kept in an oven at 240 °C for 4 days. Finally the dark brown slurry was filtered and washed with DDW until the filtrate is neutral. The solid catalyst was dried at 120 °C overnight before use.

Characterization of the Catalysts

[0051] The synthesized catalysts were characterized with powder X-ray diffraction, nitrogen sorption methods, FESEM, TEM/ HRTEM, TGA, and FT-IR. The process for heterogeneous and catalyst stability test was done by analyzing the catalyst treated under reaction conditions by XRD.

Example 2

Catalysts Activity Testing

[0052] The catalytic activity testing was performed in a ½" open ended quartz tubular fixed bed flow reactor at atmospheric pressure. The activity of the catalyst was tested at different reaction temperatures. First, the catalyst mixture was prepared by mixing 80 mg of catalyst, 40 mg of soot, and 400 mg of SiC powder. The catalyst/ soot weight ratio was 2:1. Prior to testing the catalyst, soot and SiC were mixed in a mortar with a spatula in order to provide the loose contact between the catalyst and soot. All the gases used in this study were of ultra high purity (UHP) and were purchased from Airgas East Inc. (Salem, NH). The catalysts were pretreated in flowing He (20 mL/min) at 100 °C for 1 hour to completely dry and remove any adsorbed species on the surface. Application relevant tests were performed simulating the typical diesel soot engine exhaust conditions. A gas mixture including 10% O₂, 500ppm (0.05%) NO₂, balanced by

argon was passed through the fixed bed reactor, at a flow rate of 15,000 L/ kg cat-hour. As such, while the temperature was increased according to a temperature programmed oxidation procedures, the amount of CO₂, and CO discharged from the outlet was by a SRI 8610 C gas chromatograph (GC) equipped with a 6' molecular sieve, a 6' silica gel column, and a thermal conductivity detector (TCD).

Example 3

Catalytic Results

[0053] The catalysts were tested for their activity in soot oxidation under real diesel engine exhaust gas compositions (10 % oxygen + 500 ppm NO₂). The contact between the catalysts and soot was considered as a loose contact. The activity of the catalyst at different temperatures and their kinetics, the selectivity of the final product towards carbon dioxide, and the catalyst stability under diesel soot exhaust conditions were studied.

Example 4

Soot Conversion Percentage Evaluation

[0054] FIG. 1 graphically shows the effect of temperature on the soot oxidation activity for noncatalytic (NON), Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM).

[0055] The activity upon soot conversion percentage was tested with non catalytic soot oxidation, commercial manganese oxide, undoped cryptomelane, and cobalt doped cryptomelane catalysed soot oxidation. Cobalt doped cryptomelane showed a significant catalytic activity compared to the other catalyzed and non

catalyzed reactions. The soot conversion started between 150 – 200°C and the oxidation rate increased drastically between 250 – 300°C. The 100 % soot conversion was achieved at 349°C, which is very low compared to the reported values. The reaction was continued till 450°C. Between 349 - 450°C the catalysts continued showing its maximum activity and 100% soot conversion was achieved.

[0056] FIG. 2 shows the temperatures at which 10% (T10), 50% (T50), 90 % (T90), and 100% (T100) were achieved with non catalytic soot oxidation and the reactions catalyzed by different catalysts. Cobalt doped cryptomelane achieved 100% conversion at a temperature which is 275°C lower than the temperature at 100% conversion with no catalyst. The commercial manganese oxide, on the other hand did not achieve 100% conversion even at 475°C. Compared to commercial manganese oxide, cobalt doped cryptomelane achieved 90% conversion at a temperature which is 132°C lower than that for the commercial material.

[0057] FIG. 2 shows the oxidation temperature of soot with noncatalytic (NON), Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM).

[0058] FIG. 3 graphically shows activation energies for non-catalyzed (NON), and catalyzed [Co-doped cryptomelane (Co-OMS-2), hydrothermally synthesized undoped cryptomelane (HY-OMS-2), and commercially available manganese oxide (COM)] soot oxidation reactions. FIG. 3 graphically shows the activation energy comparison between non catalyzed reaction and catalyzed reactions. Cobalt doped cryptomelane shows the lowest activation energy which is 24.9 kJ/mol lower than that of non-catalyzed reaction.

[0059] FIG. 4 graphically shows selectivity towards carbon dioxide as a function of temperature for non-catalyzed (NON) and Co-doped cryptomelane catalyzed

(Co-OMS-2) reactions. FIG. 4 graphically shows the selectivity towards carbon dioxide. Co doped OMS-2 produces 100% carbon dioxide during the oxidation of soot which can be considered as a huge improvement. The selectivity remained at 100 % throughout the experiment. No signal in the gas chromatograph was observed for carbon monoxide. On the other hand, non catalyzed soot oxidation achieved a maximum selectivity of 50% towards carbon dioxide at 350°C. The selectivity then fluctuated in between 35 – 45%.

[0060] FIG. 5 shows XRD patterns for the reaction mixture consists of Co- doped cryptomelane (Co-OMS-2), soot, and silicon carbide, at different reaction temperatures. The catalyst stability was tested by analyzing the XRD at different temperatures under reaction conditions (FIG. 5). In the XRD patterns peaks for both the catalyst and SiC were observed. The peaks for catalyst were obtained at different reaction temperatures, (25- 350°C) under reaction operating conditions. The peak positions of the obtained XRD patterns remained the same, indicating the crystal structure stability of the catalyst under reaction conditions. Hence, the catalyst prepared in the present disclosure can be successfully regenerated and recycled.

[0061] All patents and patent applications, test procedures (such as ASTM methods, UL methods, and the like), and other documents cited herein are fully incorporated by reference to the extent such disclosure is not inconsistent with this disclosure and for all jurisdictions in which such incorporation is permitted.

[0062] When numerical lower limits and numerical upper limits are listed herein, ranges from any lower limit to any upper limit are contemplated. While the illustrative embodiments of the disclosure have been described with particularity, it will be understood that various other modifications will be apparent to and can be readily made by those skilled in the art without departing from the spirit and scope of the disclosure. Accordingly, it is not intended that the scope of the claims appended hereto be limited to the examples and descriptions set forth

herein but rather that the claims be construed as encompassing all the features of patentable novelty which reside in the present disclosure, including all features which would be treated as equivalents thereof by those skilled in the art to which the disclosure pertains.

[0063] The present disclosure has been described above with reference to numerous embodiments and specific examples. Many variations will suggest themselves to those skilled in this art in light of the above detailed description. All such obvious variations are within the full intended scope of the appended claims. Also, the subject matter of the appended dependent claims is within the full intended scope of all appended independent claims.

WHAT IS CLAIMED IS:

1. A method for oxidizing soot from diesel exhaust gas from a diesel engine, said method comprising:
 - providing a diesel particulate filter for receiving the diesel exhaust gas;
 - coating a catalyst composition on the diesel particulate filter; wherein the catalyst composition comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2); and
 - contacting the soot from the diesel exhaust gas with the catalyst coated diesel particulate filter at a temperature sufficient to oxidize the soot to carbon dioxide.
2. The method of claim 1, wherein the contacting is conducted at a temperature of about 350°C or less.
3. The method of claim 1, wherein the contacting is conducted at a temperature of the diesel exhaust gas.
4. The method of claim 1, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.
5. The method of claim 1, wherein the doped manganese oxide octahedral molecular sieve (OMS-2) comprises cobalt doped manganese oxide octahedral molecular sieve (OMS-2).
6. The method of claim 5, wherein the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.
7. The method of claim 1, wherein the catalyst composition to soot weight ratio is from about 1.5:1 to about 3:1.

8. The method of claim 1, wherein the oxidation of the soot produces no carbon monoxide, and the catalyst composition contains no noble metals or rare earth metals.
9. The method of claim 5, wherein the cobalt in the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) is present in an amount of from about 0.1 weight percent to about 25 weight percent.
10. The method of claim 1, further comprising regenerating the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) at a temperature and for a period to time sufficient to substantially oxidize the soot to carbon dioxide.
11. A diesel exhaust gas treatment system comprising: a diesel particulate filter for receiving diesel exhaust gas from a diesel engine and collecting soot; and a catalyst composition coated on the diesel particulate filter; wherein the catalyst composition comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).
12. The system of claim 11, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.
13. The system of claim 11, wherein the doped manganese oxide octahedral molecular sieve (OMS-2) comprises cobalt doped manganese oxide octahedral molecular sieve (OMS-2).
14. The system of claim 13, wherein the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.
15. The system of claim 11, wherein the catalyst composition to soot weight ratio is from about 1.5:1 to about 3:1.

16. The system of claim 11, wherein oxidation of the soot produces no carbon monoxide, and the catalyst composition contains no noble metals or rare earth metals.

17. The system of claim 13, wherein the cobalt in the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) is present in an amount of from about 0.1 weight percent to about 25 weight percent.

18. The system of claim 13, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) is regenerated at a temperature and for a period to time sufficient to substantially oxidize the soot to carbon dioxide.

19. A method of providing a catalyst composition on a diesel particulate filter, said method comprising: providing a diesel particulate filter; and applying a catalyst composition for facilitating soot oxidation on the diesel particulate filter; wherein the catalyst comprises a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

20. The method of claim 19, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.

21. The method of claim 19, wherein the doped manganese oxide octahedral molecular sieve (OMS-2) comprises cobalt doped manganese oxide octahedral molecular sieve (OMS-2).

22. The method of claim 21, wherein the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.

23. The method of claim 19, wherein the catalyst composition to soot weight ratio is from about 1.5:1 to about 3:1.

24. The method of claim 19, wherein the oxidation of the soot produces no carbon monoxide, and the catalyst composition contains no noble metals or rare earth metals.

25. The method of claim 21, wherein the cobalt in the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) is present in an amount of from about 0.1 weight percent to about 25 weight percent.

26. The method of claim 19, further comprising regenerating the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) at a temperature and for a period to time sufficient to substantially oxidize the soot to carbon dioxide.

27. A catalyst composition for facilitating soot oxidation, said catalyst comprising a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

28. The catalyst composition of claim 27, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.

29. The catalyst composition of claim 27, wherein the doped manganese oxide octahedral molecular sieve (OMS-2) comprises cobalt doped manganese oxide octahedral molecular sieve (OMS-2).

30. The catalyst composition of claim 29, wherein the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) has a Cryptomelane type structure.

31. The catalyst composition of claim 27, wherein the catalyst composition to soot weight ratio is from about 1.5:1 to about 3:1.

32. The catalyst composition of claim 27, wherein oxidation of the soot produces no carbon monoxide, and the catalyst composition contains no noble metals or rare earth metals.

33. The catalyst composition of claim 29, wherein the cobalt in the cobalt doped manganese oxide octahedral molecular sieve (OMS-2) is present in an amount of from about 0.1 weight percent to about 25 weight percent.

34. The catalyst composition of claim 27, wherein the doped or undoped manganese oxide octahedral molecular sieve (OMS-2) is regenerated at a temperature and for a period to time sufficient to substantially oxidize the soot to carbon dioxide.

ABSTRACT OF THE DISCLOSURE

A method for oxidizing soot from diesel exhaust gas from a diesel engine. The method involves providing a diesel particulate filter for receiving the diesel exhaust gas; coating a catalyst composition on the diesel particulate filter; and contacting the soot from the diesel exhaust gas with the catalyst coated diesel particulate filter at a temperature sufficient to oxidize the soot to carbon dioxide. The catalyst composition is a doped or undoped manganese oxide octahedral molecular sieve (OMS-2) material. A diesel exhaust gas treatment system that includes a diesel particulate filter for receiving diesel exhaust gas from a diesel engine and collecting soot; and a catalyst composition coated on the diesel particulate filter. The catalyst composition is a doped or undoped manganese oxide octahedral molecular sieve (OMS-2).

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Fig. 1

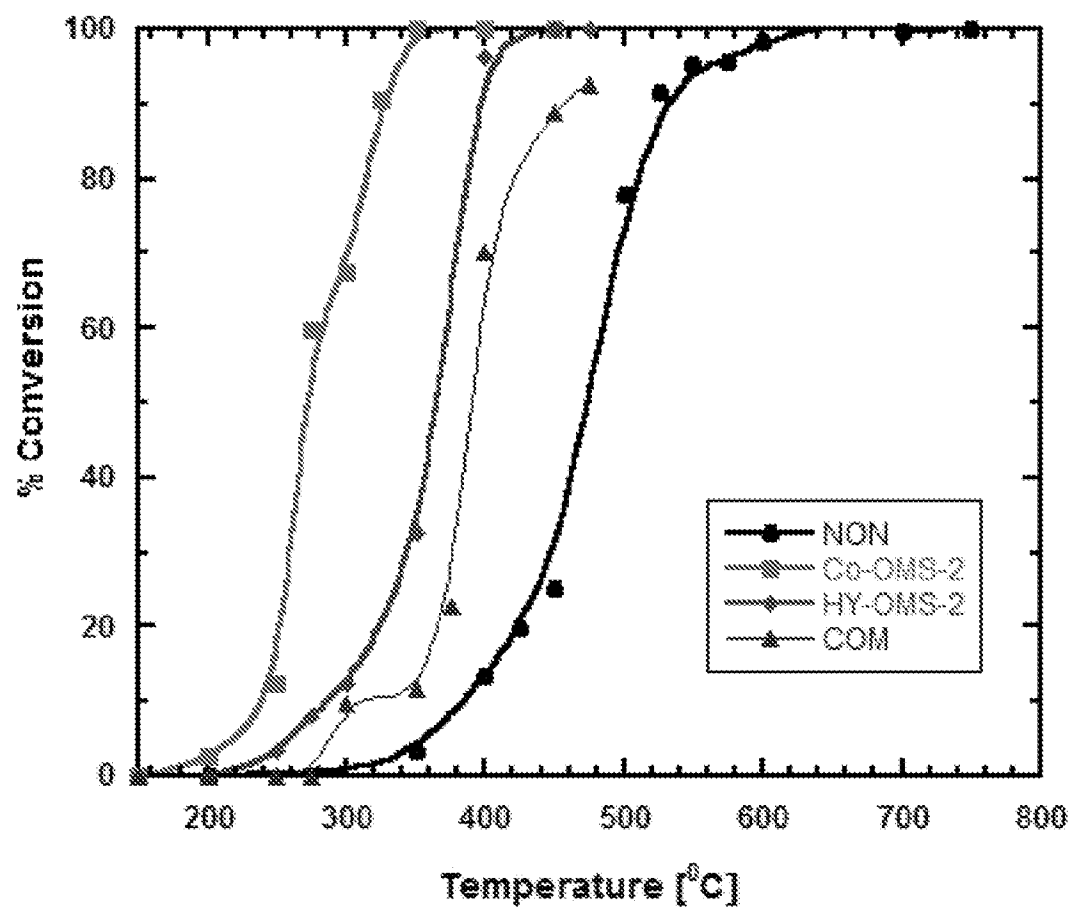
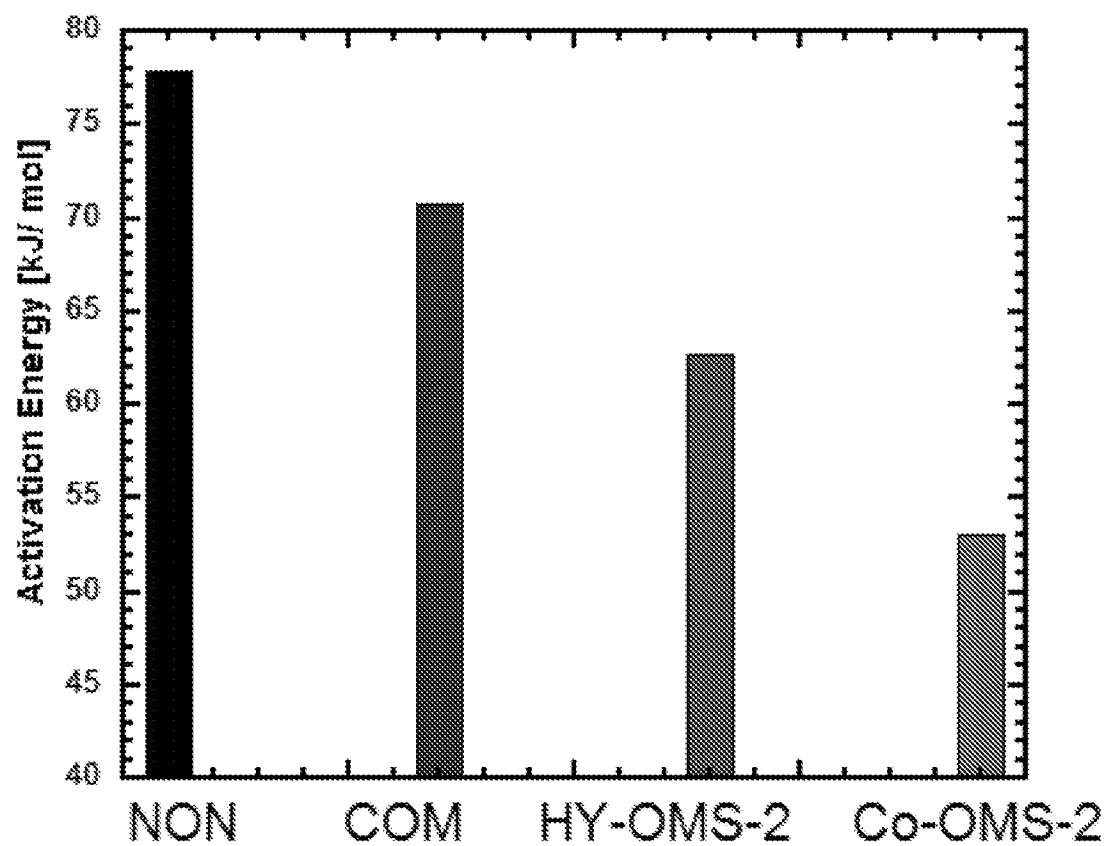


Fig. 2

	T10	T50	T90	T100
NON	382	475	532	626
Co-OMS-2	238	270	328	349
HY-OMS-2	284	368	394	430
COM	332	393	460	NA

NA = Not achieved

Fig. 3



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Fig. 4

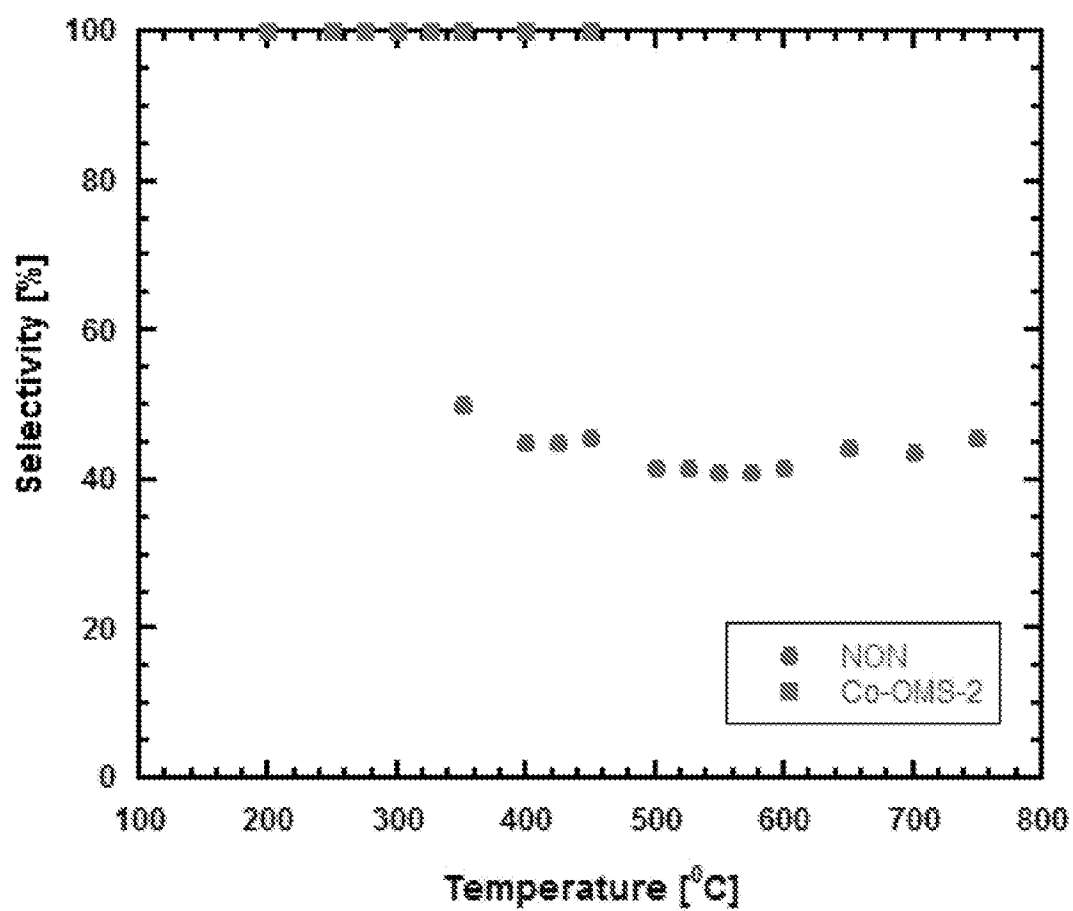


Fig. 5

