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Fuel Properties and Oxidation Stability of Jatropha Biodiesel produced from Microwave Assisted Transesterification Using Renewable Calcium Oxide as Heterogeneous Catalyst.

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ABSTRACT

Heterogeneous catalysts are promising catalysts for optimal biodiesel yield from transesterification of vegetable oils. In this work calcium oxide (CaO) heterogeneous catalyst was synthesized from eggshell. Calcination was carried out at 800°C for 5h and characterized using Fourier transform infrared spectroscopy. Catalytic efficiency of CaO was tested in transesterification of Crude Jatropha Oil (CJO). A biodiesel yield of 87% was estimated at 1.75wt % catalyst, 5 min reaction time and 60°C temperature. The density, flash point, viscosity,acid value, iodine value, cloud points, pour points, water content and fatty acid composition of biodiesel were determined and compared to ASTM and EN standard. The physical properties of the biodieselwere agreed with the standards. The fatty acid predominantly composed of 15-Octadecanoic acid, Heptadecanoic acid and 14-methyl-8-hexadecy with a percentage composition of 22.31%, 10.58% and 10.03% respectively. The biodiesel produced had low unsaturated fatty acids 0.40% and the saturated fatty acid was 22.31% which indicate good oxidation stability.

Keywords: *Biodiesel, Jatrophacurcas oil, Transesterification, Heterogeneous catalyst, eggshell, Gas Chromatography-Mass Spetrophotomer (GCMS)*

1. Introduction

Energy is for accessing the level of human economic development globally(Motasemi and Ani 2012).Petroleum is presently the most dominant source of global energy supply. The consumption of the petroleum-derived fuel is on the increase, due to economic increase and humanpopulation.Petroleum-derived fuel prices are not stable and the combustion of this fuel are greatly contributing to environmental pollutionThe concernof air pollution and global warming resulting from the combustion of fossil fuels drove people away from fossil-derived fuels to the production and use of environmentally friendly (panahi, 2013). Biofuelsare renewable fuel derived from biomass. Biofuel types include biogas, bio-alcohol and biodiesel. Biodiesel has gained attention as a renewable, biodegradable, non-toxic, and environmentally friendly alternative to petroleum diesel due to its lower emission of green-house gases. (Lee *et al*., 2009).

Biodiesels are promising alternative fuels for diesel engines due to their environmental and strategic advantages (Ani and Elhameed, 2014).Biodiesel can be produced via transesterification of triglycerides (vegetable oil) with methanol using homogeneous basic or acidic catalysts. However, the use of homogeneous catalysts causes many problems such as reactor corrosion, soap formation, difficulty in reuse of catalysts, and the production of large amount of waste water, which increase the overall cost for biodiesel production.In order to overcome this draw backs,the production of biodiesel using heterogeneous base catalyst has been receiving more attention, because of ease of catalyst separation from the reaction mixture, catalyst re-usabilityand low energy-consumption(Canakciand Gerpen, 2015).Different types of heterogeneous catalyst such as metal oxides, zeolites, hydrotalcites, Cao and γ-alumina, have

been used for the production of biodiesel from various edible and non edible vegetable oil (Bokhari, 2015).

Calcium oxide has attracted a special interest due to its low cost, high activity, and abundance in nature (Buasri and Loryuenyong, 2015).One of the best methods for obtaining CaOis the thermal
decomposition of calcium carbonate ($CaCO_3$) present in minerals such as calcite or animal products like eggshells(Win, and Khine, 2016),Oyster and *Pyramidella*Shells (Buasri*et al.*, 2015), waste Shells of Mussel, Cockle, and Scallop(Buasri*et al.*, 2013), shells of razor and surf clam, crab shell, or pork bone,Mussel, cockle, andscallop. CaOof very high purity could be obtained from renewable sources by waste valorization from other industries such as the food industry. Different researchers have reported the application of calcium oxide for the production of biodiesel. Buasriet *al.* (2015) studied the effect of CaO catalyst on biodiesel production of JCO with a catalyst range of 2 - 6wt%. It was reported that there is a rapid yield in biodiesel as the catalyst concentration increase from 2-4%. The biodiesel yield decrease as the catalyst increase from 5-6wt%. This is due to the formation of slurries which were too viscous for adequate mixing. The optimum conditions that yielded 93% for both waste shell-derived catalysts (Oyster and PyramidellaShells), were 5min reaction time, microwave power of 800W, methanol/oil molar ratio of 15, and catalyst loading of 4wt%. The experimental results show that CaO catalyst had excellent activity and stability during reaction.Marwan and Indarti (2016) reported the biodiesel yields at various catalyst loadings 0-5wt% for the transesterification of palm oil with methanol at a 1:9 molar ratio. At initial stage the biodiesel rapidly increased from catalyst range of 0-3wt% to give biodiesel yield of 55.6%, whereas the maximum yield was 59.2%, using 4wt% catalyst. However as the catalyst loading increased rom 4.0wt% to 5.0wt%, biodiesel yields continued to decrease This was due to the lack of mass transfer at higher catalyst loadings.

Ani and Elhameed (2014)reported that the best yield of 96% was obtained using microwave exit power of 300W,with a molar ratio of oil to methanol 1:6, 8wt% CaO derived from eggshell waste and the optimum reaction time of 7 min. The yields of biodiesel was decreasing by increasing the reaction time,within the catalyst concentrations range of 3 - 8 % microwave exit power and this is due to increasing the reaction temperature. Although at higher catalyst concentration of 8%wt, biodiesel yields are higher but by increasing the reaction time the biodiesel yields will reduced.

Microwave assisted transesterification of non-edible oil has been employed for the production of biodiesel from different oil bearing seeds.Different researchers have reported the application of microwave and calcium oxide (CaO) for the production of biodiesel:Buasri*etal.* (2015) reported the effect of power during the transesterification of jatropha oil between 180–800W. The jatropha biodiesel yield were 150W(26.58%), 300W(45.84%), 450W(57.37%), 600 W (75.90%), and 800 W (93.92%) for oyster shell. It was observed that the higher microwave power gave rise to higher biodiesel yield. The optimum conditions, which yielded a conversion of oil of nearly 93% for both waste shell-derived catalysts, were reaction time 5min, microwave power 800W, methanol/oil molar ratio 15, and catalyst loading 4wt%.It was considered that the related chemical reactions are accelerated by microwave energy, giving rise to intense localized heating and thereby accelerating the chemical reaction and giving high product yields in a short time.

Joshi *etal.* (2016) reported the effect of MW powers from 300W- 900W. The yields of algal biodiesel were found to increase from 45.6% -61.4%; Jatropha biodiesel 41.8%-58.0%; whereas, the yields of Pongamia biodiesel were obtained 39.8% - 55.3%. To obtain the maximum yields of biodiesels using optimum MW power, experiments were performed using 5.0 g of oil, 3wt% of Cao catalyst, 12:1 methanol:oil with a reaction time of 3min. The biodiesel yields were initially increased with MW power from 300W to 700W; however, alcoholysis reactions at 800W and 900W have shown negative effect on biodiesel yields. It was observed that the reaction yield increases with increase in MW irradiation power; however, if the MW power is too high it may degrade the triglycerides to FFA and also makes highly disordered and drastic molecular interactions which has some adverse effect on biodiesel yields. Therefore, 700W of microwave power is considered as the optimum MW power for the present studies.The aim of this study was to determine the fuel properties and oxidation stability of biodiesel produced from microwave assisted transesterification of JCO using egg shell derived calcium oxide.

2.METHODOLOGY

2.1 Materials

Jatropha Curcasoil was obtained from chemical Engineering Department Federal University of Technology Minna. Egg shell was obtained from Minna. Methanol was purchased from a Panlac chemicals in Minna, Niger State.

2.2 Method

2.2.1 Catalyst Preparation

The solid heterogeneous catalysts were prepared by a calcination methodin which the egg shells waste were washed with distilled water to remove unwanted particles and oven-dried at low temperature, and the dried waste shells calcinedat 800°C in a muffle furnace for 5 h The solid result was crushed and sieved to pass 100–200 mesh screens. The products (38–75 μm) were obtained as white powder. All calcined samples were kept in the close vessel to avoid their reaction with carbon dioxide (CO_2) and humidity in air before being used. The CaO catalysts were obtained as white. This catalyst contains $CaCO_3$ which is converted to CaO after calcination at 800∘C temperatures for 5h

2.2.3 Transesterification of JCO

The transesterification was carried out in a 500 ml glass reactor equipped with sensor and mechanical stirrer, placed inside a household microwave oven.The fixed 70ml of JCO and the 1.75wt% CaO catalysts was added to the reactor. Then the methanol was introduced to the oil at constant methanol/oil molar ratios of 12:1. The reaction was operated at 50-60°C with varied reaction time of 5-20min under microwave irradiation. After the reaction was completed the sample was allowed to stand at room temperature for 5 min; the reaction mixture was poured inside the separating funnel. Two layers were formed wherein the upper layer was yellow in color including mixtures of crude ethyl ester, while the bottom layer was dark brown containing glycerol, the crude ethyl ester was separated and the excess glycerol (residual catalyst and methanol) washed with 70ml warm distilled water twice in a separator funnel.The ester phase was placed in a glass cylinder to allow the excess water dry off at room temperature. The washed ethyl ester was then oven dried

2.2.4 Characterization of biodiesel

The biodiesel produced was slightly washed with warm distilled water and then characterized to determine its properties. The following parameters were determined

Determination of Moisture Content

5g of the sample was weighed into a weighed crucible. The crucible and sample taken were then transferred into the oven set at 100°C index and allowed to dry overnight. At the end of the 24 hours the crucible plus sample were removed from the oven and transferred to the desiccator and cooled for 10 minutes and weighed.

Given that,

The weight of empty crucible = W_O
Weight of crucible + sample = W1
Weight of crucible + oven dried sample = W3

$$\% \, Moisture \; Content = \frac{W3 - Wo}{W1 - Wo} \times 100 \qquad (1)$$

Determination of Specific Gravity:A 50 ml neatly dried empty bottle was measured, its weight taken as x_1. Distilled water was used to fill the bottle, weighed and reported as x_2. Same density bottle was used but the water was substituted with biodiesel sample after drying, the weight of the bottle with biodiesel was recorded as x_3, then the specific gravity was evaluated using the following equation,

$$Specific \; Gravity = \frac{x_3 - x_1}{x_2 - x_1} \qquad (2)$$

Determination of Density: This was determined by measuring the oil in a 250 ml graduated cylinder and inserting into the cylinder a known mass of an hydrometer to note if it floats in the oil or it can be calculated from a known specific gravity of the oil with weight of water (1000 g) and then the density was calculated using equation 2

$$Density = \frac{Weight \; of \; oil}{Volume \; of \; oil} \qquad (3)$$

Determination of Cloud Point: Biodiesel was poured into a fixed level jar for a test; it was then put into a hot water bath. The measured temperature where the biodiesel start to form cloud around the base of the jar now becomes the FAME cloud point (Gerpen and canakci 2001).

Determination of Pour Point: Biodiesel sample was kept in a freezer at a temperature of 50 ^{0}C until it solidified. The pour point now became equal to the base temperature in the jar at which the biodiesel started to gel (Lapuerta*et al.*, 2008).

Determination of Kinematic Viscosity:A viscometer with a third spindle was placed inside a beaker filled with hot biodiesel of 40 ^{0}C and allowed to stay for a period of about 30 min with the speed of the spindle set on automatic. The biodiesel was allowed to flow freely until the meniscus moved from the initial marked time to the final mark and the readings displayed on the screen of the viscometer. A stop watch was used for the timing, this step was repeated three more time, the mean value was recorded and that gave the viscosity of the biodiesel (Lapuerta*et al.*, 2008).

Determination of Flash Point: Biodiesel was placed in a closed container and flame was burnt on it. A thermometer was used to observe the temperature value at which the flame burnt to the end with a pop sound. This process was repeated up to four times and the average was taken which gave the flash point(Verma *et al*, 2016).

Determination of Acid Value and Free Fatty Acid: Two grams of the oil was dissolved in 50 ml of the neutral solvent in 250 ml conical flask, 3 – 4 drops of phenolphthalein indicator was then added and titrated against 0.1 M KOH the content was constantly shaked until a pink colour which persists for fifteen seconds was obtained

$$Acid\ value = \frac{Titre\ value \times 0.1\ M\ KOH \times 56.10}{Weight\ of\ sample(g)} = mgKOH/g \quad (4)$$

$$FFA = \frac{Acid\ value}{2} = mgKOH/g \quad (5)$$

Determination of Iodine Value

Iodine value measure the degree of unsaturation in vegetable oils. This value of oil or fat is defined as the weight of iodine absorbed by 100parts by weight of the sample. The iodine value is mostly used for identification of oil or to assign a particular group to the oil. The common method of determining iodine value is Wijis' method. 0.25g of oil was weighed into iodine flask and it was dissolved into 10ml of chloroform. 25ml of Wiji's iodine solution was mixed vigorously and allowed to stand in a dark corner for exactly 30minute. The excess iodine was determined by addition of 10ml of 15% potassium iodide solution; 100ml of water was added and titrated with 0.1M normal sodium thiosulphate using starch as indicator. The titration was continued until the blue black colour just disappeared after vigorous shaking. A blank determination was also carried out without the oil with the same condition.

$$Iodine = \frac{(b-a)\times 1.29}{Weight\ of\ sample} \quad (6)$$

3. Results and Discussion

3.1 Fuel properties of biodiesel

Table 1: Fuel properties of Jatropha curcas oil, biodiesel and EN(Knothe et al 2006)

Properties	Unit	Jatropha curcas oil	Biodiesel	EN 14214	
Moisture	%	5.26	0.2	>0.05	
Acid Value	Mg/KOH/g	8.9	Trace	<0.01	
FFA		4.45	-	>0.5	
Flash Point	°C	95	105	>101	
Cloud Point	°C	14	0.9	-	
Pour point	°C	6	-2	-	
Viscosity 28°C Viscosity 40°C	mm^2/sec	5.4 3.8	2.2 3.7	1.9-6.0 3.5–5.0	
Density	Kg/m^3	947	900	860-900	
Specific	Kg/m^3	954	974		

Gravity					
Iodine		23	31.93	>120	

The qualitative characteristic of biodiesel is an important index for determining it commercial viability. Density is one of the important properties of biodiesel which influences the efficiency of the fuel atomization for air less combustion systems (Salles*et al.*, 2010).The density of the biodiesel in this study is 900 kgm^{-3}. According to ASTM D6751 and EN14214 standards, the density of biodiesel is between 860kg/m^3– 900kg/m^3 for biodiesel.This result is consistent with the upper limit of ASTM D6751 and EN14214 standards.The value is however higher than the findings of Marwan and Indarti (2016) and Buasri*et al.* (2015) who reported that the densities of biodiesel from palm oil and Jatropha curcasoils were 888 kg/m^3 and 879 kg/m.Enweremadu*et al.* (2011) reported that variation in biodiesel densitiesare principally associated with the difference in short chain aliphatic acid (fatty acid) composition of biodiesel and the level of impurity in the raw vegetable oil used as feedstock.

Flash point is the minimum temperature at which the fuels will ignite when exposed to flame. A high value of FAME flash point shows that there is no unreacted methanol in the sample(Terigar*et al.*, 2010). The flash point for biodiesel is set at above 101 ° C and 130 ° C in the respective European standard (EN 14214) and ASTM 06571. The result obtained in this work for biodiesel produced using egg shell derived CaO catalyzed was 105° C and are quite consistent with this standards. This shows that the biodiesel produced is essentially free from methanol as even small amount of methanol can reduce the flash point greatly (Terigar*et al.*, 2010).

Viscosity is one of the important properties that determines the resistance to flow. High viscosity can lead to poor atomization during fuel spraying and can cause engine deposits, and incomplete combustion. The viscosities for biodiesel at 28 ° C (EN 14214) and 40 ° C ASTM 06571are 1.9-6.0 mm^2/s and 3.5-5.0 mm^2/s, respectively. The viscosities obtained in this work for biodiesel was 2.2 mm^2/s and 3.8 mm^2/s for 28°C and 40°C respectively and are quite consistent with this standard. The viscosity of 5.4 mm^2/s of JCO used as feedstock was lowered to 2.2 mm^2/s for biodiesel produced, and that confirms the transesterification process was effective. This values lies within these standards and quite in agreement with the findings of Marwan and Indarti (2016) who reported that the viscosity of palm oil methyl ester is 4.5 mm^2/s by using sea shell derived CaO catalyst. The result also shows appreciable consistency with the findings of Buasri*et al.* (2015) who reported the viscosity of Jatropha curcasoil methyl ester is 4.5 mm^2/s by using oyster derived CaO catalyst.

Acid number is the mass of KOH in milligrams that is required to neutralise the acidic constituents in one gram of biodiesel sample. Acid value is use to measures the content of free fatty acids contained in biodiesel sample. According to EN 14214 Standard, acid values must be less than 0.01. There is a trace of acid value in this work, which shows that the JCO contain less FFA (%wt), because high FFA (%wt) causes soap formation during alcoholysis process and lead to difficulties in separation of biodiesel from its by-product; as a result it reduced the yield or conversion.

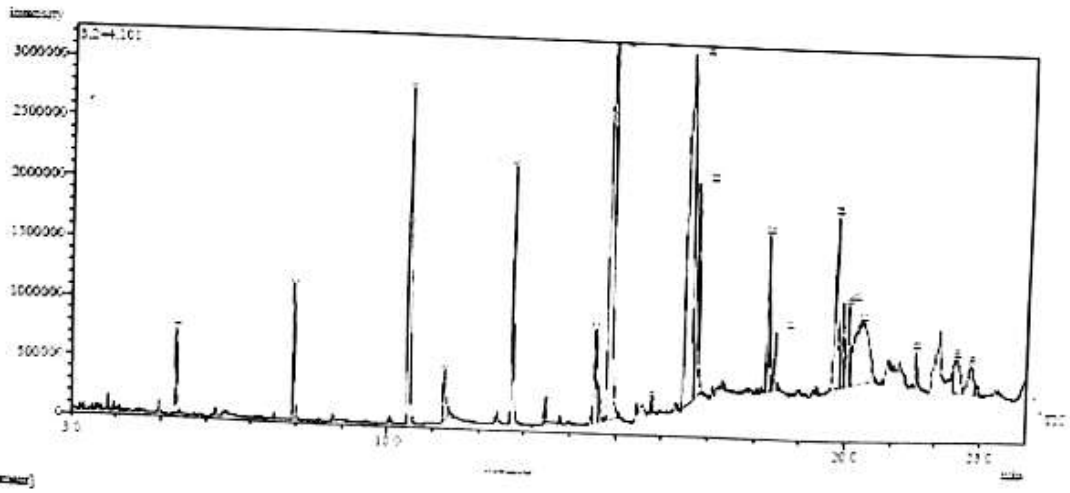
The iodine value greatly influences fuel oxidation and deposits formed in diesel engines injectors (Salouaet *al.*, 2010).The iodine value for this study was determined to be 31.93. Iodine values are used to classify oils as either drying oil (>130), semi-drying oil (100-130) and non-drying oil (<100) .Vegetable oil with iodine value between 100 and 130 belongs to the groups

of semi-drying oil. This group of oil absorbs atmospheric oxygen slowly; produce only soft film after prolonged exposure to air (Alfa, 2010). The iodine value of *JCO* suggests their use in production of alkyl resin, shoe polish, varnishes.

The cloud points is defined as that temperature in which a cloud or haze of wax crystal appears at the test when the oil is cooled under prescribed condition. The cloud point is also the temperature at which wax first becomes visible when the fuel is cooled (Singh and Singh, 2010).The cloud points for this study for renewable Cao catalysts is 0.9 °C. The values obtained in this study is lower than the report of 11 °C byBuasri and Loryuenyong (2017) and Ania and Elhameed (2014) with cloud points of 6°C. The low cloud points from this study is an indication that the fuel will performs satisfactory even in cold climate conditions since the tendency for gel formation is low, because higher cloud point can affect the engine performance and emission adversely under cold climate conditions.

The pour points obtained for biodiesel was – 2 °C; this values falls within the ASTM standard – 15 to 10 °C. The results obtained also shows appreciable consistency with the earlier report of 0 °C by Bokhari*et al.*(2015) and - 1°C Balat, (2011) but differs with result of Buasri*et al.* (2015) and Buasri and Loryuenyong (2017) who reported 9 °C and 8 °C respectively. The low values from this study are due to the higher content of unsaturated fatty acid in raw Jatropha curcas oil. Although the value was found to be within specified limit but biodiesel from Jatropha oil was suitable not only for the tropical region but also for moderate temperature region. When the ambient temperature is below the pour point of oil, waxes precipitates out and loose their flow characteristic. This wax can block the filters and fuel supply line. Under this condition the fuel cannot be pumped through the injector.

The water content of the biodiesel was 0.2, this values lies within the biodiesel standards. According to EN14214 standards, water content is >0.05 for biodiesel standard. This values lies within these standards and quite in agreement with the findings of Buasri*et al.* (2015) who reported that the water content of palm oil methyl ester is (0.02) by using renewable CaO catalyst



3.2 Fatty Acid Composition

Figure 1. GC-MS graph for Jatropha Biodiesel

Table 2: Fatty acid composition of jatropha seed oil biodiesel

Lines	Molecular Formula	Fatty Acid	Structure Number	Area (%)
1	$C_9H_{18}O_2$	Octanoic acid	C9:2	1.49
2	$C_{11}H_{28}O_2$	Myristic acid	C11:2	2.33
3	$C_{13}H_{26}O_2$	Dodecanoic acid	C13:2	8.96
4	$C_{12}H_{24}O_2$	Lauric acid	C12:0	1.47
5	$C_{15}H_{30}O_2$	Lauric acid	C15:2	5.75
6	$C_{20}H_{40}O_2$	Arachitic acid	C20:0	0.37
7	$C9H_{20}O$	Octano	C9:0	2.64
8	$C_{17}H_{34}O_2$	Heptadecanoic	C17:2	18.11
9	$C_{14}H_{28}O_2$	Myristic acid	C14:0	0.40
10	$C_{19}H_{36}O_2$	15-Octadecanoic acid	C19:2	22.31
11	$C_{19}H_{38}O_2$	linoleic	C19:2	5.30
12	$C_{12}H_{26}O_2$	Octano	C12:2	3.90
13	$C_{21}H_{42}O_2$	Eicosanoic	C21:2	1.24
14	$C_{19}H_{36}O_2$	Octadecadienoyl Chloride	C19:2	6.18
15	$C_{12}H_{26}O$	Decanoic	C12:0	2.56
16	$C_{23}H_{46}O_2$	Docasanoic acid	C23:2	2.24
17	$C_{17}H_{32}O$	14-methyl-8-hexadecy	C17:2	10.03
18	$C_{28}H_{56}O_2$	Heptacosanoic	C28:2	0.63
19	$C_{14}H_{30}$	Tridecane	C14:0	2.29
20	$C_{21}H_{44}$	Heptadecane	C_{21}:0	1.80

Table 2 showsthat the predominantly fatty acid in biodiesel are 15-Octadecanoic acid,Heptadecanoicacid and 14-methyl-8-hexadecy with a percentage composition of 22.31%, 10.58% and 10.03% respectively. Jatropha biodiesel of this work had low unsaturated fatty acids0.40% and the saturated fatty acid is 22.31% which is high. This shows that biodiesel with high percentage of saturated fatty acid have good oxidation stability.The findings from this study show appreciable agreement with the report of Echim*et. al.* (2012) that biodiesel from vegetable and animal origin containing highly saturated methyl esters have oxidation stability. Fatty acid without double bonds in their carbon chain is referred to saturated fatty acid; some of these are Lauric acid, arachitic acid, octanoic acid, decanoic acid, tridecane acid, heptadecane and Myristic acid having 12.0,20.0, 9.0, 12.0, 14.0, 21.0 and 14.0, carbon atoms respectively. Poly-unsaturated fatty acids have either two or three double bonds in their carbon chain, such asOctanoic acid, Myristic Acid,Dodecanoic Acid, LauricAcid,Heptadecanoic Acid, Octadecanoic Acid, linoleic acid, Octano, Eicosanoic Acid, Octadecadienoyl Chloride, Docasanoic Acid, 14-Methyl-8- hexadecy Acid, Heptacosanoic Acid, having 9.2, 11.2,13.2,

15.2, 17.2,19:2, 12.2 , 21.2, 19.2, 23.2, 17.2, and 28.2 carbon atoms. Various studies on fatty acids and the resulting esters shows that saturated fatty acids result in esters with higher cetane number, higher oxidation stability and higher lubricity while unsaturated fatty acids result in esters with better cold flow whereas polyunsaturated fatty acid result in low oxidative stability and high freezing points (Singh *et al.*, 2009).

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