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Full Length Research Paper

Use of nanotechnology for the production of biofuels from butchery waste

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Biodiesel can be used as a substitute for fossil fuels and successful studies have been carried out in different applications. The advantages with biodiesel are lower bulk, higher energy density, as well as more transportable and storable capability. We used butchery waste for the production of hydrocarbon gases and biodiesel. For this purpose we used nano catalyst and anatase form of titanium dioxide photocatalyst at room temperature. In the first experiment, butchery waste was cracked into low melting point oil, solid residue and hydrocarbon gases. In the second experiment, cracked oil was transesterified to biodiesel with NaOH at room temperature and atmospheric pressure. The results yielded best quality biodiesel. The economics of this novel process is much more cost competitive due to cheap raw material (butchery waste) that contains high levels of fatty acids. Photocatalysis gave hydrocarbons of prime importance. This study reports an interesting finding that butchery waste could be used for not only the production of biodiesel but also for hydrocarbons. This technology differs from others in that it uses low energy input, cheap and reusable catalyst, with low sulphur and nitrogenous waste gases than petro-diesel and is environment friendly.

Key words: Butchery waste, photocatalyst, biofuel, cobalt nanoparticles, nickel nanoparticles, transesterification.

INTRODUCTION

Economical, environmental and energy security concerns resulting from excessive reliance on petroleum are forcing countries throughout the world to search in the form of ethanol and biodiesel (Farrell et al., 2006). The development of alternative and renewable fuels has become a very intense research area in the last decade. Among these use of biodiesel derived from the transesterification of vegetable oils, animal fats and waste frying oils has increased significantly in many countries around the world. One of the most common methods used to reduce oil viscosity is transesterification. Which in the production of a fuel comprised of mono-alkyl esters of long chain fatty acids called biodiesel. Biodiesel is the most widely accepted alternative fuel for diesel engines due to its technical, environmental and strategic advantages (Enweremadu et al., 2011). Moreover biodiesel is technically competitive with conventional, petroleum-derived diesel fuel and requires virtually no changes in the fuel distribution infrastructure. Other advantages of biodiesel as compared to petro-diesel include reduction of most exhaust emissions, biodegradability, higher flash point, inherent lubricity (Haas et al., 2001) and the fact that it is of domestic origin.

With the increasing demand of biodiesel, it is imperative to reduce burdens on the edible oil and to synthesize biodiesel from waste non-edible fatty acids to prevent pollution and economize the biodiesel production. Biomass has been gaining intense attention as a renewable energy source, due to world petroleum demand and concerns over the environmental impact of CO_2 production. Compared with fossil fuels, biomass has

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a number of advantages, such as low emissions of SOx and NOx, CO_2 neutral, renewable, as well as locally produced. Thermal conversion processes of biomass include direct combustion, pyrolysis, gasification and liquefaction. Fast pyrolysis of biomass is considered to be a promising thermo-chemical process in which biomass is converted to pyrolytic oil, char and gas (Zhang et al., 2011).

In Pakistan waste animal fats are cheap and easily available from the butcher shop particularly during the Muslim festival of Eid-ul-Azha. Moreover, there is no renewable use of animal fat and lot of fats are thrown in garbage as waste or people take them for their pet animals. Animal fats are primarily water-insoluble, hydrophobic made up of triglycerides (Sonntag, 1979). Fatty acids vary in carbon chain length and in the number of saturated/unsaturated bonds. The higher stearic and palmitic acid contents give beef tallow the unique properties of high melting point and high viscosity. Gasification of waste animal fat by using Ni and Co nano catalyst was useful (Mahmood et al., 2010). Thermal decomposition of biomass with catalyst shows two advantages, firstly primary gases like CO, CO_2, CH_4 and soot, etc., are formed. Secondly, on reforming, other hydrocarbons are formed (Davidian et al., 2006). Gates (2000) have employed molecular nano particles in catalysis.

The benefits of applying nanotechnology to catalysis include improved activity, lifetime, resistance to poisoning and other novel abilities (Mahmood and Hussain, 2010; Mahmood et al., 2010).

These improvements and novelties cannot always be achieved with catalysts prepared by other methods. An example of expanding the catalytic capabilities of regular catalysts through nano scale manipulation is cobalt-based catalysis. Cobalt nano particle catalysts may be influenced by their size and structure and by other additional components, such as Si, Ni and Mg (Yang et al., 2001; Kesavan et al., 2001; Son et al., 2002; Hussain et al., 2009). Cobalt nano particles, in particular being cheap, need mild reaction conditions for high yields of products in short reaction times as compared to the traditional catalysts (Mahmood and Hussain, 2010; Mahmood et al., 2010).

Nickel catalyst is widely used in gasification (Davidian et al., 2006; Kimura et al., 2006; Swierczynski et al., 2007; Svoboda et al., 2008; Hussain et al., 2009). The melting point of nano particles is size dependent. On mixing nano particles of Ni with Co, catalytic ability is increased (Guo et al., 1995). Properties of nano particles can be studied by transmission electron microscope (TEM), scanning electron microscope (SEM) and X-ray diffraction (XRD) (Niasari et al., 2008). Ni nano particles can be used in catalytic reduction of aromatic and other carbonyl compounds (Kidwai et al., 2006; Mahmood and Hussain, 2010; Mahmood et al., 2010).

Nickel nano particles are used (Kidwai et al., 2006) as an efficient and selective catalyst for the reduction of aldehydes. The Ni nano particles catalytically play significant role in the reduction of aldehydic groups in the presence of other functional groups, such as $–NO_2$, –CN and double bonds at α and β positions. Besides Ni, Co also plays a significant role in changing the chemistry of fatty acid. It is evidently shown that Co nano particles (Nianqiang et al., 2004) stretched the band of the carboxyl group of pure liquid and leads to the formation of two new bands with characteristic of the asymmetric trans (COO-) and the symmetric cis (COO-) stretch.

Catalytically pyrolysed oil was transesterified and also converted into biodiesel by photocatalyst. Photocatalyst are used to produce surface oxidation that will help to eliminate organic compounds or nearby bacteria, when it is exposed to the sun or fluorescent lamp. By applying this principle to water treatment, dissolving NOx in the air or room air purification, photocatalyst can be used for various steps in purifying a contaminated environment.

Photocatalyst is also very useful in energy production from biomass. Stamate and Lazar (2007) reported that in the absence of a catalyst active substances, oxidation of most hydrocarbons proceeds slowly, which can be explained by kinetic arguments. A photocatalyst decreases the activation energy of a given reaction. In the result of photo-induced processes, often particles with strong oxidation and reduction ability occur. Titanium dioxide based anatase shows the highest photocatalytic activity (Mahmood et al., 2010). For removal of phenol, titanium oxide based photocatalyst was used (Suryaman et al., 2009). A wide range of organic compounds are converted into water and carbon dioxide. This behavior of photocatalyst is responsible for killing pathogenic microorganisms.

The goal of this study was to use solid butchery fatty waste for production of biodiesel. For this purpose, catalytic pyrolysis and transesterification photocatalytic conversion of fats into biodiesel were used.

MATERIALS AND METHODS

Production and characterization of Co and Ni nano particles

The cobalt chloride and nickel chloride ($CoCl_2$. $6H_2O$ and $NiCl_2$. $6H_2O$) were purchased from Sigma Aldrich USA, and 1,10-phenanthroline was purchased from Fluka. All solvents were of analytical reagent grade and were used without further purification. For the synthesis of complex of cobalt, 0.5 M (molar) solution of 1,10-phenanthroline and 0.5 M (molar) solution of cobalt chloride were separately prepared in 1-propanol. The 1,10-phenanthroline solution was taken in dropping funnel and very slowly dropped to the cobalt chloride solution with constant stirring at a temperature of 40 to 50°C. The pink precipitate of cobalt/1,10-phenanthroline complex appeared after almost one third (1/3) of 1,10-phenanthroline solution was added to cobalt chloride solution, the adding of the 1,10-phenanthroline solution to the salt solution was continued till complete precipitate formed in the reaction mixture.

The precipitate was then filtered and washed two times with 1-propanol to remove the un-reacted 1,10-phenanthroline/cobalt chloride. The precipitate was dried under infrared (IR) lamp and then under vacuum. The complex was taken in a two-neck flask and was kept in tube furnace for decomposition. One side of the flask was connected to argon cylinder and the other was used as outlet for argon and decomposition gases. The temperature of the furnace was raised to 500°C at a heating rate of 0.5°C min^{-1} and the contents were kept at 500°C for 24 h and then allowed to cool to room temperature under an inert atmosphere of argon gas to yield Co nano particles. Same procedure was used for the preparation of Ni nano particles. The particle morphology and size was studied by scanning electron microscope (SEM-Zeiss Supra 50VP with EDS Oxford), transmission electron microscope (TEM-FEI Tecnai F20 S-TWIN 200 FEG) and X-ray diffraction (XRD). The X-ray diffraction pattern of the particles powders were collected with PANalytical, Netherlands, diffractometer (Model 3040/60 X^{l} pert PRO) equipped with a Cu Kα radiation source. Using Scherrer formula, based on line broadening, the mean crystal sizes of the powders were determined (Niasari et al., 2008; Mahmood and Hussain, 2010; Mahmood et al., 2010).

Photocatalyst production and characterization by TEM and XRD

For the preparation of polycrystalline nano particles of TiO_2 Fluka $TiCl_4$ (98% purity, analytical grade) was diluted up to 15% (1.33 M) in 15% H_2SO_4 in distilled water. A light yellow colored solution was obtained with pH of -0.75. The solution was stirred for 2 h at room temperature. The NaOH solution (3 M) was added drop wise (controlled through high performance liquid chromatographic (HPLC) pump). The resulting solution was continuously monitored for pH. When the pH reached -0.11, the light yellow coloration disappeared and transparent solution was formed. The drop wise addition of NaOH was continued until the pH of the resulting solution becomes 0.85. At this pH white precipitate was obtained. The pH of the white precipitate was further increased to 2 by the addition of NaOH. At this pH, the reaction was stopped. It was then washed with distilled water to remove all the NaOH solution. The material was dried overnight at 100°C. The TiO_2 powder thus obtained was calcined at 500°C for 6 h. The particle morphology and size was studied by transmission electron microscope (TEM-FEI Tecnai F20 S-TWIN 200 FEG) and XRD. The X-ray diffraction pattern of the particles powders was collected with PANalytical, Netherlands, diffractometer (Model 3040/60 X^{l} pert PRO) equipped with a Cu Kα radiation source. Using Scherrer formula, based on line broadening, the mean crystal sizes of the powders were determined (Mahmood et al., 2010).

Collections of samples for oil preparation

Waste animal fat samples were collected from slaughter house, Islamabad, Pakistan. Solid fat 728 g heated at about 110°C to remove the moisture present in the fat. The sample leftover (546 g) of sample was left, which contains triglycerides. This sample was then heated in the furnace in a round bottom flask in the presence of Co and Ni nano particles. 100 g of solid fat was taken in a round bottom flask and 0.1 g of Ni and Co nano particles are added in the conical flask and heated in a furnace. The temperature of furnace was increased gradually, at 250°C, vapours start to come out from the flask. Furnace is heated up to 400°C for 10 to 15 min and then, it was switched off. During heating, match stick was brought near the outlet, flashes of fire are formed which indicated the presence of fuel gases. These gas samples were analyses on gas chromatography-mass spectrometry (GC-MS). Results showed hydrocarbons like (CH_4 and C_2H_2). After heating the sample left in the flask, that contains 57.5% liquid with high viscosity, 18.75% solid fat and 23.75% mixture of different gases (CH_4, C_2H_2, etc).

Biodiesel formation by transesterification

Biodiesel is produced using base catalyzed transesterification, as it is the most economical process requiring only low temperatures and pressures and producing 98% yield. The catalyst that is used is sodium hydroxide. Three grams of NaOH were dissolved in 90 ml CH_3OH using hot plate and magnetic stirrer, and then 30 g oil was added in it. The whole solution was left in the electrical shaker for overnight mixing. The temperature was maintained at 45°C with shaking speed at 110 rpm. The 52.5% liquid (biodiesel) with low viscosity was obtained along with 18.9% soap and 28.2% solid residues which contain glycerin. Liquid obtained from this experiment was diluted with chloroform (in 1:5 ratios). The liquid extract was filtered before analyzing on Fourier transform infra red spectroscopy (FTIR) (Thermo-Nicolet Nexus 670 Spectrophotometer). Standard petro-diesel and biodiesel samples were collected from Pakistan State Oil (PSO) filling station of Islamabad. FTIR spectrum of standard petro-diesel, standard biodiesel and of sample after transesterification were analyzed (Mahmood and Hussain, 2010).

Small fatty acids and hydrocarbons formation by photocatalysis

Liquid extract of animal fat 30 g was taken in a round bottom flask and 0.3 g photocatalyst (Titanium dioxide) was added to it. Then, this solution is placed in sunlight with continuous stirring for 3 h. Liquid obtained from this experiment was diluted with chloroform (in 1:5 ratios). The liquid extract was filtered before analyzing on gas chromatograph (GC) (Model Varian CP-3800) (Mahmood et al., 2010).

RESULTS AND DISCUSSION

Energy plays a pivotal role in socio-economic development by raising the standard of living. Biomass has been used as an energy source for thousands of years by humankind. In this study, solid fatty butchery waste of animal origin was converted into biofuels like biodiesel and hydrocarbons. Nickel is a widely distributed metal that is industrially applied in many forms. Evidence shows that Ni nano particle posses many new characteristics, which include a high level of surface energy, high magnetism, low melting point, high surface area and low burning point. Therefore, it can be widely used in the catalytic decomposition in modern industries. Pyrolysis is the thermal decomposition of solid biomass at high temperature and pressure (Navarro et al., 2009). The thermal decomposition produces hydrocarbons at 1000°C and absence of air. The catalytic pyrolysis decreases reaction temperature up to 650°C. The simplest type of decomposition is the fixed bed counter current decomposition. The major advantages of this type of decomposition are its simplicity, high charcoal burnout

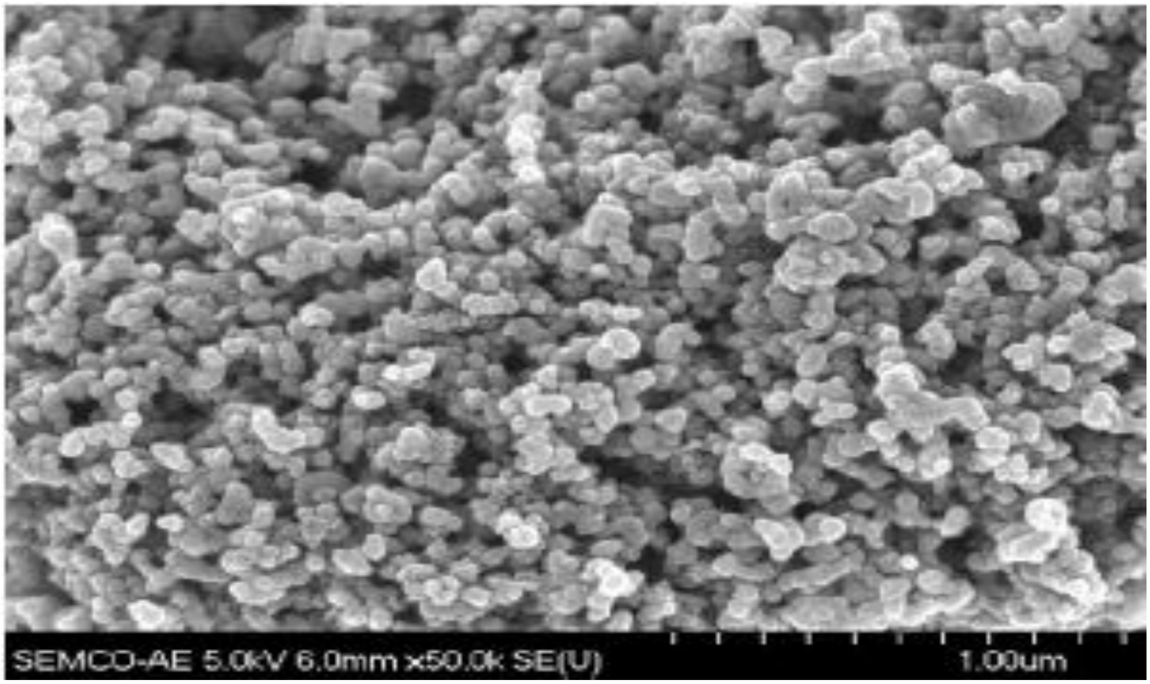

Figure 1. SEM image of Co-nano particles (Mahmood and Hussain, 2010; Mahmood et al., 2010).

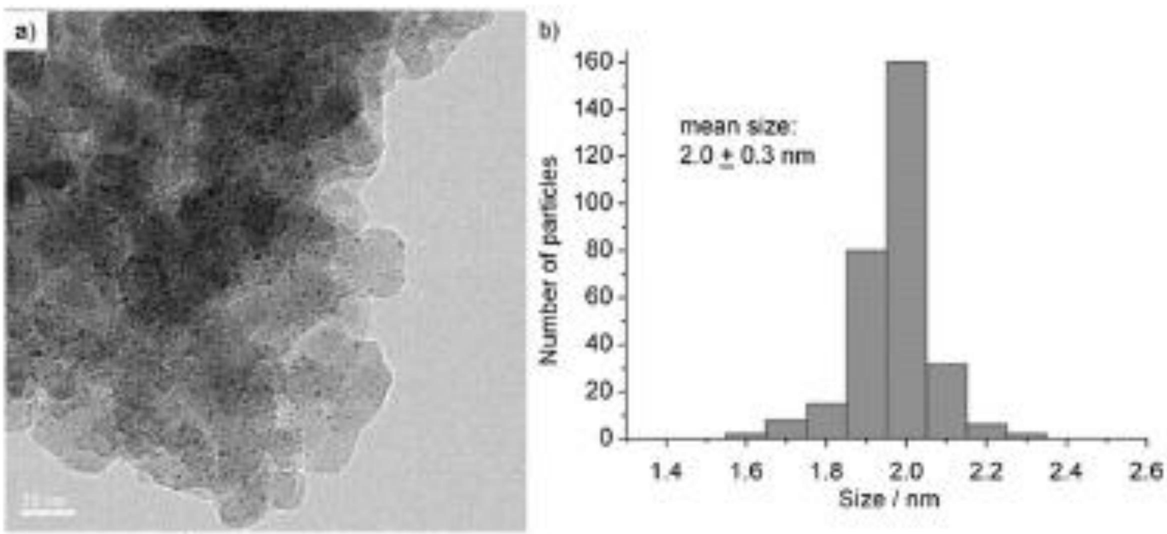

Figure 2. TEM image of Ni-nanoparticles (Mahmood et al., 2010).

and internal heat exchange that lead to low gas-exit temperatures and high decomposition efficiencies (Quaak et al., 1999).

For cobalt and nickel particle's characterization, SEM, TEM and XRD techniques were used as reported (Niasari et al., 2008). The SEM and TEM images are given in Figures 1 and 2. Their average particle sizes (Ni = 4.2 nm, Co = 7.2 nm and TiO_2 = 3 nm) were calculated by using the procedure described by Figures 1 and 2 demonstrate the morphology as well as crystallite size of metallic nano particles which are synthesized through precipitation method. These images indicate that the particles are uniform, regular and spherical sponge like in shape in both the cases. Cobalt and nickel fall in the size range of 2 to 10 nm. This is comparable to the crystallite size calculated from XRD by applying Sherrer

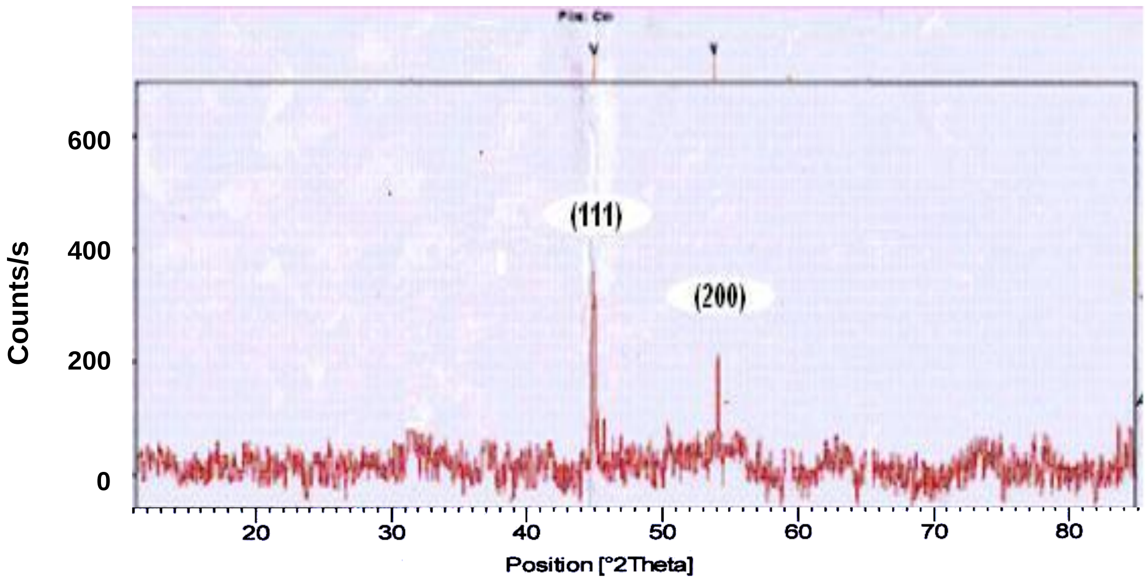

Figure 3. XRD spectrum for Co nano particles (Mahmood and Hussain, 2010; Mahmood et al., 2010).

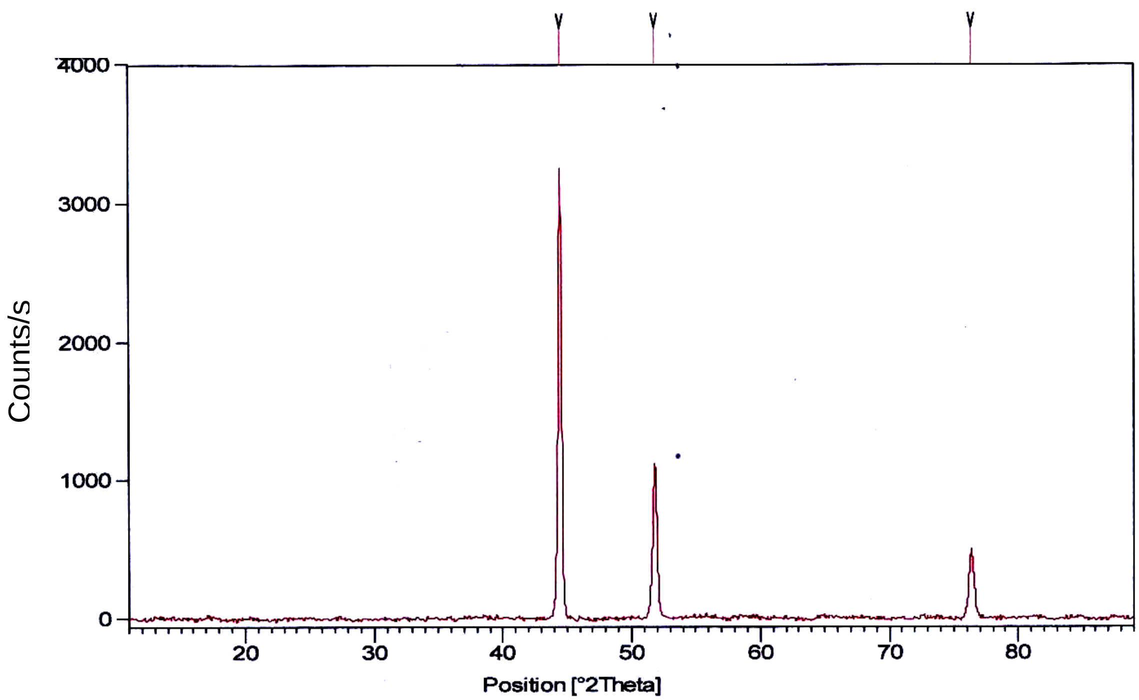

Figure 4. XRD spectrum for Ni nanoparticles (Mahmood et al., 2010).

formula. The XRD (Figures 3 and 4) has given prominent peaks for the metallic nano particles of nickel and cobalt. The XRD pattern of fresh cobalt nano particles was studied from the data; cubic structure for cobalt nano particles was obtained by using the standard American Society for Testing Minerals (ASTM) XRD files. The XRD peaks (Figure 3) corresponds to the indices (111) and (200). The peaks obtained for nickel nano particles (Figure 4) are consistent with the indices (111), (200) and (222) of pure face cantered cubic with 2Θ at

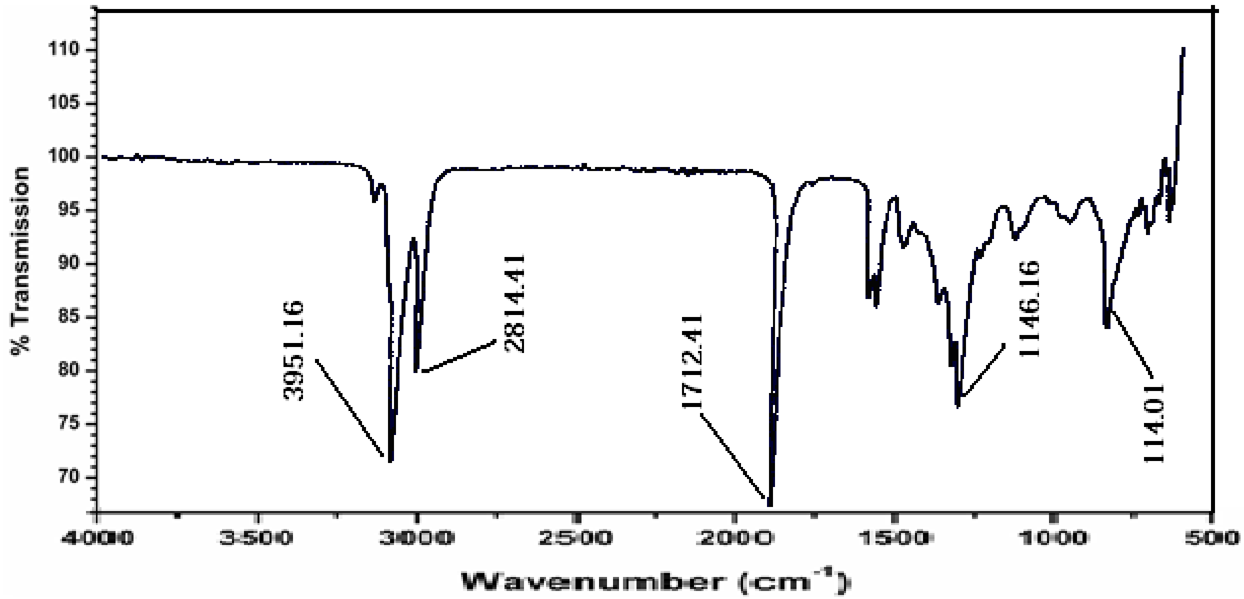

Figure 5. FTIR spectrum peaks area (in cm^{1}) for standard biodiesel.

44.4, 51.8 and 76.4°, respectively. From the afore-mentioned indices, it could be concluded that the nano particles of nickel prepared through this method were pure with a controlled phase of face cantered cubic (FCC) structure.

The Ni nano particles act as a green catalyst for selective reduction of the aldehydic group in the presence of other functional groups, namely: $–NO_2$, –CN and alkenes to give the corresponding alcohols in excellent yields (Kidwai, 2006). These alcohols react with fatty acids to produce esters. High temperature and high activity of Co and Ni cracked long chains to smaller ones. The samples of standard petro-diesel, standard biodiesel and of sample after transesterification were analyzed by FTIR. The comparison of these three samples showed similarity.

Figures 5, 6 and 7 are the FTIR spectrum of standard biodiesel, petro-diesel and biodiesel produced from animal fat, respectively. Table 1 is showing comparison of data of these three diesels. Table 2 shows vibrational mode assignments of FTIR spectrum peaks area (in cm^{-1}). The assignments of peaks are listed in Table 2. The broad feature between 3346 and 2816 cm^{-1} is undoubtedly due to the O-H stretch of the carboxylic acid. No other functional group had such a broad and intense bond at high wave number. Two sharp bonds at 2915.86 and 2816.55 cm^{-1}, which were superimposed on the O-H stretch, were attributed to the asymmetric CH_2 stretch and the symmetric CH_2 stretch, respectively. The intense peak at 1558.82 cm^{-1} was derived from the existence of the C=O stretch, and the band at 1020.69 cm^{-1} exhibited the presence of the C-O stretch (Vijaya et al., 2002). The O-H in-plane and out-of-plane bonds appeared at 1401.38 and 937 cm^{-1}, respectively (Smith, 1999).

The FTIR spectra of standard petro-diesel, standard biodiesel and that of biodiesel produced from animal fat are studied comparatively. Our biodiesel samples are very much similar to that of standard. It may be due to the compounds that have almost the same chemical groups. However, some differences are detectable. The position of the carbonyl band in FTIR is sensitive to substituent effects and to the structure of the molecule (Pasto et al., 1992). The methoxy carbonyl group in biodiesel shows a different bond position of the C=O vibration when compared to the carbonyl bond in our sample. The peak of this bond changed from 1451 cm^{-1} in standard diesel to 1732 cm^{-1} in standard biodiesel to 1558 in our samples. Some new bonds were also observed in our samples that may be the effect of Ni and Co nano particles; because they have high reactivity and they may affect the chemistry of animal fat.

After the preparation of TiO_2 based photocatalyst, TEM was used for its characterization. Figure 2 showed TEM spot like structures with maximum size of 3 nm. Graig et al. (2009) used titanium dioxide to convert a mixture of carbon dioxide and water vapors to methane. They reported higher yield of methane than previously reported (FuturePundit: Nanotubes for Photocatalysis Produce Methane, 10th July, 2009), (http://www.futurepundit.com /archives/006019.html) (Mahmood et al., 2010). By using sun light energy, titanium dioxide converted CO_2 and moisture to methanol and ethanol. These alcohols may be

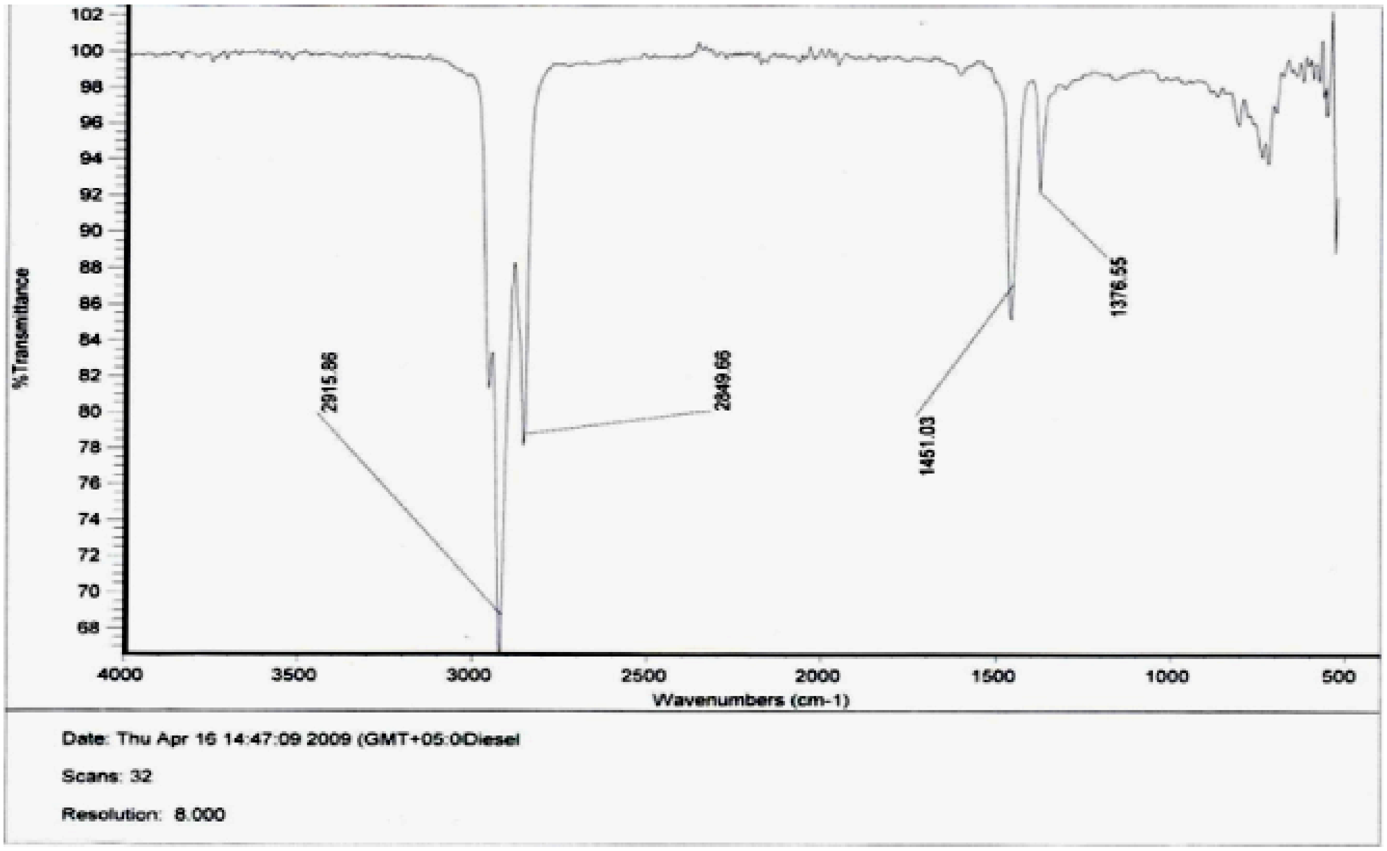

Figure 6. FTIR spectrum peaks area (in cm1) for standard petro-diesel.

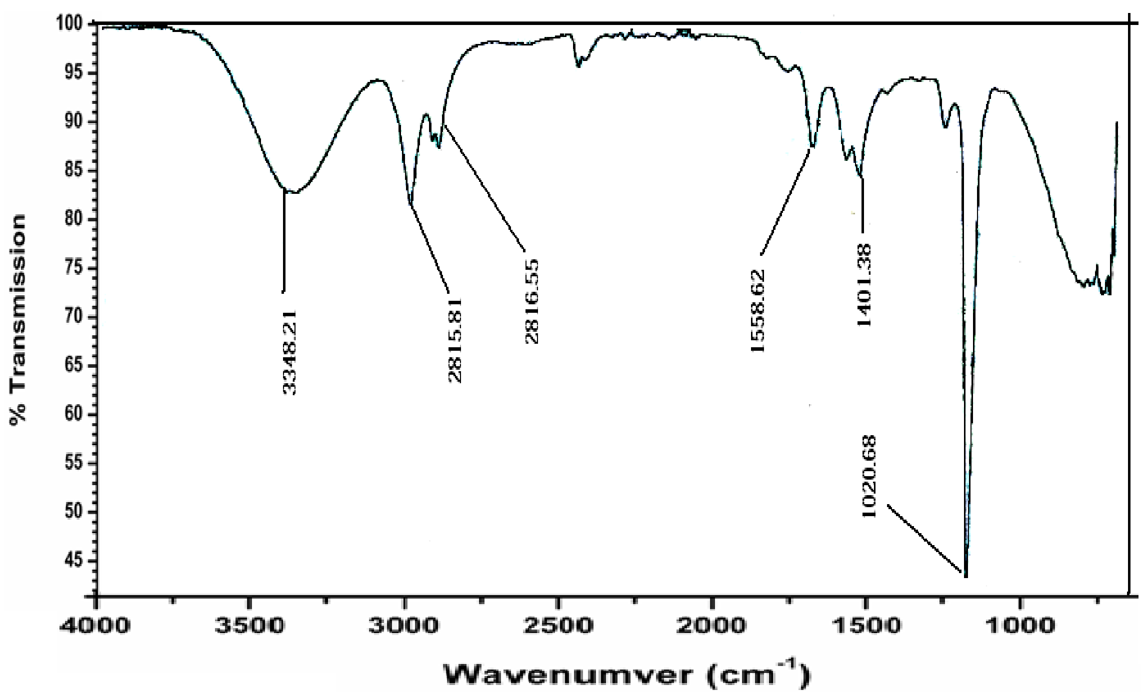

Figure 7. FTIR spectrum peaks area (in cm^{-1}) biodiesel from animal fat.

Table 1. Comparison of FTIR spectrum peak area data (in wave number cm^{-1}).

Petro-diesel (cm^{-1})	Biodiesel standard (cm^{-1})	Biodiesel from animal fat (cm^{-1})
2915.86	2924.14	2915.86
2849.66	2849.66	2816.55
1451.03	1732.41	1558.62
1376.55	1169.66	1401.38
-	714.48	1020.79

Table 2. Vibrational mode assignments of FTIR spectrum peaks area (in cm^{-1}).

Peak (cm^{-1})	Assignment
3500 - 2500	O-H stretch
3005	C-H stretch in C=C-H
2924	asymmetric CH_2 stretch
2854	symmetric CH_2 stretch
1710	C=O stretch
1462	in plane O-H band
1409	CH_3 umbrella mode
1285	C-O stretch
937	out-of-plane O-H stretch
715	CH_2 rocking

oxidized to produce fatty acids and may be reduced to produce alkanes. The temperature and high photocatalytic activity of titanium dioxide cracked long chains to smaller ones.

Photocatalyst are strong oxidizing agents. They create electronic holes in order to break the organic matter to carbon dioxide and water in the presence of light. The photocatalytic properties of TiO_2 were discovered by Akira Fuji Shima in 1972 and the process on the surface of TiO_2 was called Honda-FujiShima effect. TiO_2 is a semi conducting material which can be chemically activated by light. Anatase shows the highest photo activity under UV light. The benefits of applying nanotechnology to catalysis include improved activity, lifetime, resistance to poisoning and other novel abilities. These improvements and novelties cannot always be achieved with catalysts prepared by other methods. Titanium dioxide, also known as titania is the naturally occurring oxide of titanium. You will find TiO_2 in all kinds of paint, printing ink, plastics, paper, synthetic fibers, rubber, condensers, painting colours and crayons, ceramics, electronic components along with food and cosmetics. Many studies have been published on the use of TiO_2 as a photocatalyst for the decomposition of organic compounds. The TiO_2 is active under UV light. Photocatalytic activity (PCA) is the ability of a material to create an electron hole pair as a result of exposure to ultraviolet radiation. The resulting free-radicals are very efficient oxidizers of organic matter. Photocatalytic activity in TiO_2 has been extensively studied because of its potential use in sterilization, sanitation, and remediation applications. The ability to control PCA is important in many other applications utilizing TiO_2, including paint pigments and cosmetics that require low PCA. The oxidative behaviour of photocatalyst oxidizes hydrocarbons to alcohols. The -OH radical produced by anatase can decompose a variety of organic compounds. Titanium dioxide (TiO_2) has been regarded as an excellent photocatalyst because of its performance, low cost, no toxicity, stability, wide band gap and availability. UV illumination of titanium dioxide leads to the formation of powerful agents with the ability to oxidize and decompose many types of bacteria, organic and inorganic materials. In the following, the principles and potential applications of TiO_2 photo-catalysis were discussed (Stamate and Lazar, 2007). Graig A. Grimes, professor of electrical engineering and his team used titanium dioxide to convert a mixture of carbon dioxide and water vapors to methane (Graig et al., 2009). Using outdoor, visible light, they reported a 20-times higher yield of methane than previously published attempts (FuturePundit: Nanotubes for photocatalysis Produce Methane, 10 July, 2009), (http://www.future pundit.com/archives/006019.html) (Mahmood et al., 2010). It is suggested that methane might be raw material for producing larger alkanes, esters and alcoholic compounds. Perhaps nano-materials will provide efficient means to capture solar energy for this purpose too. The higher activity, smaller size and larger surface area of photocatalyst are responsible oxidation of carbon based biomass to carbon dioxide. The moisture content helped the preparation of methane, methanol and ethanol. Jones et al. (2007) supported this idea of photocatalysis. Results of photocatalysis show the presence of small chain fatty acid along with traces of n-heptane and n-octane. Out of these two, n-octane play a significant role in determining the quality of petroleum products, while n-heptane also have significant role in industry. Heptane (and its many isomers) is widely applied in laboratories as a totally non-polar solvent. A liquid is ideal for transport and storage. In the grease spot test, heptane is used to dissolve the oil spot to show the previous presence of organic compounds on a stained paper. It is done by shaking the stained paper in a heptane solution for about half a minute. The *n*-heptane is the zero point of the octane rating scale. It is undesirable in petrol, because it burns explosively, causing engine knocking, as opposed to branched-chain octane isomers, which burn more slowly and give better performance. But on the other hand it may be an important component of biodiesel which have low capacity to burn and have low knocking capacity by adding the n-heptane in it. We can increase the efficiency of the biodiesel. However n-octane is used in organic syntheses, calibrations and azeotropic distillations and is a common component of gasoline and other petroleum products. The engine fuel ant knocking properties of an isomer of n-octane (2,2,4-trimethylpentane or iso-octane) are used as a comparative standard in the octane rating system.

Conclusions

In this study, the following conclusions are drawn:

1. Solid butchery waste fatty material is a good tool for cheap industrial production of biodiesel.
2. After catalytic pyrolysis and transesterification solid fatty residue, is rich in N, P and proteins can be used as bio-fertilizer.
3. Solid butchery waste is an excellent source of environment friendly hydrocarbon fuel gases.

The use of this solid waste as a source of biofuel will add green energy technology in the existing environment. The process would be ideal for countries where it is produced and consumed.

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