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THE EFFECT OF PYROLYSIS TEMPERATURE AND TIME, ON THE PROPERTIES OF POLYETHYLENE WAX AND HYDROCARBON GASES PRODUCED FROM WASTE POLYETHYLENE SACHETS

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ABSTRACT

The effects of temperature and pyrolysis time on the properties of polyethylene wax and gaseous products produced from waste polyethylene water sachets were investigated. Waste polyethylene water sachets were pyrolysed at temperatures between 110°C and 150°C and different time. The effect of temperature and pyrolysis time is significant on the yield, melting point and penetration degree of polyethylene wax produced. The polyethylene wax obtained has a penetration degree of 1-40.6 mm; the melting point was 76°C - 142°C while the yield of polyethylene wax obtained was 95.31-50.44%. Waste sachets pyrolysed at 130-150˚C for 30-40 minutes produce high quality polyethylene wax (paraffin and microcrystalline wax) from waste polyethylene water sachets with yield of above 75%, good melting point and penetration degree that meet industrial standard The remaining non condensable hydrocarbon gases produced along with the wax, which is mostly ethane, propane, propylene can be used as feedstock for the heater or sold as fuel gas.

Keywords: polyethylene wax, gaseous products, waste polyethylene water sachets, melting point, penetration degree, pyrolysis time, pyrolysis temperature.

INTRODUCTION

Municipal waste plastic represents about 8% wt. of the municipal solid waste and it generally consists of different kind of plastics: out of that polyethylene waste constitute about 30% (Brebu *et al*., 2004). Water sachet constitutes about 10% of polyethylene waste in Nigeria cities and other developing countries. This waste is difficult to be treated or recycled due to its complex nature and composition, structural deterioration of the polymeric components and the contamination with various organic, inorganic and biological residues. All over big cities in Nigeria and even in the rural communities, it is a common sight that polyethylene films, shopping bags, plastics, water sachets littering and polluting the environment. The increase of plastic waste and its harm in Nigerian environment has attracted the concern of the political and technological circles. These days the waste polyethylene (often called pure water sachets) is known to cause blockage of drainage systems in our townships and in rural commodities since these waste films are non biodegradable this has resulted in the flooding of our communities whenever it rains and subsequent destruction of houses and farmlands.

Polyethylene is a macromolecular hydrocarbon that can be converted into useful products such as oil and waxes but since the molecular chain of polyethylene is composed of $-CH_2-$, the freezing point of the diesel obtained is very high and the research octane number of gasoline is very low near 88. Thus it is not a feasible way to convert polyethylene to oil. It is, however a profitable way to convert polyethylene waste to polyethylene wax (Jixing *et al*., 2003). Waxes derived from petroleum products are basically paraffinic wax and microcrystalline wax. Waxes are used in the manufacture of candles for religious and decorative purposes, in polishes, matches, waxed paper, and cosmetics. Waxes are also generally used in the manufacture of rust preventives, crayons, rubber antioxidants, electrical insulators, paper coatings, printing inks, textile finishes, and leather dressings. Etc. (Owolabi and Amosa, 2010) and wax selection and production is determined by the type of application required.

Several studies have been carried out to produce wax from waste. A study on the conversion technology of waste polyethylene plastic to polyethylene wax was carried out by Jixing *et al*., (2003). The effect of the reaction conditions including pyrolysis temperature, pyrolysis time, and the content of the additive on the yield and quality of the polyethylene PE wax such as penetration degree and melting point were discussed. The PE wax obtained has a melting point of 104-144°C. The penetration degree is 0.1- 42.4 mm; the yield of PE wax is over 90%. Ademiluyi and Akpan (2007) studied the production of fuel oil from pyrolysis of waste water sachets. Using waste polyethylene water sachets of 97.72g at 130-190°C, they obtained fuel oil of 18.30 g, 76.14 g of wax and 3.28g of gaseous products, but the property of wax obtained was not determined in this study. This result showed that more wax can be obtained at temperatures lower than 190°C. Owolabi and Amosa (2010) studied the effective management and potentials of conversion of water sachet to a more useful product such as super wax. Four different samples wax were formulated, but only solidification time of 20-30 minutes was observed for each of the samples. Also the properties of wax such as penetration degree and melting point, tensile strength, density of the wax obtained from the waste sachets were not determined in this study. In order to produce quality wax that meets industrial standard with this polyethylene waste as shown in Tables 1 and 2, there is need to control the operating variables such as pyrolysis temperature and time during the pyrolysis process. (William *et al*, 1955).

Table-1. Showing melting point and penetration degree of different waxes for paraffin wax.

S and P Number	Melting point open cap. tube USP class II	Penetration ASTM D-1 321100/77/5
206	122-127 Deg. F	18-43
192	124-130 Deg. F	9-15
227B	128-135 Deg. F	11-16
1275	127- 135 Deg. F	11-16
173	138-144 Deg. F	10-16
673	141-146 Deg. F	10-15
434	150-156 Deg. F	10-16
674	156-163 Deg. F	10-15

Table-2. Showing melting point and penetration degree of different waxes for microcrystalline wax.

S and P Number	Melting ooint ASTM D-127	Penetration ASTM D-1 321 100/77/5
96	145-155 Deg. F	30 Max.
18	165-175 Deg. F	60-80
19	170-180 Deg. F	27-33
26	170-180 Deg. F	27-33
60	175-185 Deg. F	16-22
16	180-190 Deg.F	13-19
617	191-199 Deg. F	8-14
89	195-202 Deg. F	4 -10
624	195-202 Deg. F	4 -10

Source: Strahl and Witsch (2012)

Therefore, in this paper the conversion of polyethylene waste sachet to polyethylene wax and gaseous products would be studied. The effect of pyrolysis temperature, and pyrolysis time, on the yield, penetration degree and melting point of polyethylene wax produced from waste water sachets will be discussed.

MATERIALS AND METHOD

Materials

Batch reactor, temperature controller, waste low density polyethylene films (pure water sachet), electric heater, thermocouple, lagging materials, beaker to receive the wax and weighing balance. Polyethylene waste water sachet around the streets/ roads sides in the university campus were gathered, washed with water to remove sand and other impurities, and then dried. These waste pure water sachets were cut into pieces of about 1cm size to create higher surface area for pyrolysis. Figure-1 shows the washed, dried and waste water sachets cut into pieces. The apparatus consists of a fabricated batch reactor, with lagging for effective heat transfer, thermocouple, mercury manometer, 750ml gas cylinder was used to trap waste gases, electric heater and SHIMADZU gas chromatography to analyze waste gaseous products. The arrangement is as shown in Figure-2.

Method

100g of the pure water sachets were placed in the reactor and the reactor hopper was properly covered and tightened to prevent heat loss. The reactor was then placed on the electric heater with thermometer inserted into one of the openings on top of the reactor. Thermo-couple was also connected to the reactor to control and monitor the reaction temperature. Adequate precaution was taken to make sure that there was no leakage before commencement of the experiment. The heater was then switched on and the pyrolysis continued until the

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temperature and the residence time of the reaction is reached. Pyrolysis temperature ranging from 110°C-150°C, at different time of 20, 30, 40, 50, 60minutes were used, and the yield calculated.

Standard test method for needle penetration of petroleum wax (ASTM D 1321-97)

This test method covers the empirical estimation of the consistency of waxes derived from petroleum by measurement of the extent of penetration of a standard needle. This test method is applicable to wax having a penetration of not greater than 250mm. The sample was melted, heated to 17°C above its congealing point, poured into a container and then air cooled under controlled condition. The sample was then conditioned at test temperature in a water bath. Penetration is measured with a Penetrometer, which applies a standard needle to the sample for 5s under a load of 00g.

Figure-1. Clean and dry waste pure water sachets cut into pieces.

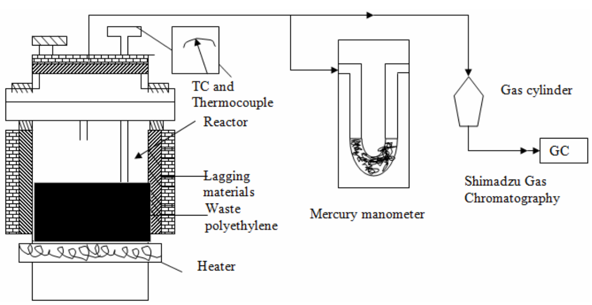

Figure-2. Reactor for pyrolysis.

Determination of melting point

Waxes are traded on the basis of melting point range. Therefore standard test method defined by ASTM (D 127-87) for drop melting point of Petroleum wax including petrolatum was used. Penetration and drop melting point test of the wax were carried out at the Nigeria National petroleum corporation's refinery laboratory at Kaduna, Nigeria. The average of the two determinations was taken as the drop melting point of the sample under test.

Gaseous products obtained at pyrolysis temperatures below 140°C

It was already observed that about 3.28% of non condensable gaseous bi-products were produced during the pyrolysis of this water sachet to produce fuel oil at a temperature above 150°C (Ademiluyi and Akpan, 2007). But the yield of these gaseous bi-products was more during pyrolysis of the waste sachets to produce wax at temperature below 140°C; hence this non condensable gaseous bi-product was carefully studied while the wax was produced. The gas collection was in the first 5 minutes as the temperature rises to 140°C with a gas bladder. There after pressure reduction was observed followed by production of dense white fumes suspected to be aromatic compounds at temperatures above 150°C. The gaseous product was sent to Shimadzu gas chromatography and was analyzed using 20ml of helium as carrier gas and at column temperature range of 40°C to 180°C. The detector was flame ionization Detector (FID) at 100°C. Flame test was carried out and a colourless blue flame was obtained.

RESULTS AND DISCUSSIONS

Waste water sachets were pyrolysed at different pyrolysis temperature and time to produced polyethylene wax. Figure-3 shows the polyethylene wax produced from waste water sachets at different pyrolysis temperature and time. As shown in Figure-1 the sachets pyrolysed were colourless but the polyethylene wax has a gray colour (Figure-3), this change in colour may be due to the ink used to print the product named on the water sachets before pyrolysis. The ink was not removed from the sachets before pyrolysing the waste sachets since Jixing *et al*., (2003) had already reported that dyes does not affect the properties of wax. The colour of wax produced from this sachet can be changed to any desired colour by mixing required dyes with the waste sachets before the pyrolysis.

The Effect of pyrolysis temperature and time on the yield of polyethylene wax

The effect of pyrolysis temperature and time on the yield of polyethylene wax is shown in Figure-4. It was observed that, at the same pyrolysis temperature, the yield decreases with the increase of pyrolysis time. At high pyrolysis temperature, the decrease in wax yield was very obvious. It can be seen from Figure-4 that, the yield was 95.31-50.44% at the pyrolysis temperature range of 110˚C-150˚C when the polyethylene waste (pure water sachet) is pyrolysed to polyethylene wax.

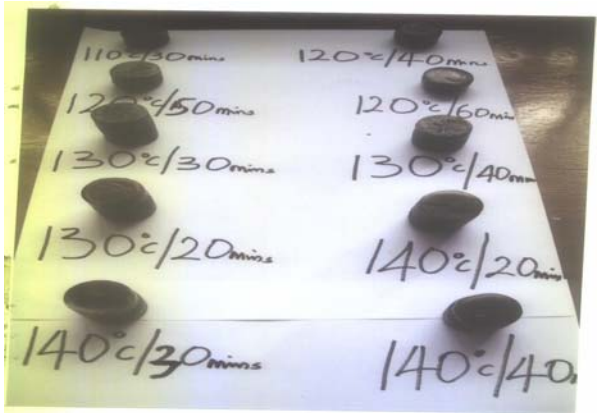

Figure-3. Polyethylene wax produced from pure water sachets showing pyrolysis temperature and time.

It was also observed that when the polyethylene waste water sachet was pyrolysed at temperature 150˚C, the yield decreases sharply from 88.10 to 50.44% as the pyrolysis time was changed from 20 to 60 minutes. At the same reaction time with an increase in pyrolysis temperature, the yield decreases. This is a very significant trend. If Polyethylene waste (pure water sachet) is pyrolysed at reaction time of 50 minutes when the temperature rises from 110˚C to 150˚C, the yield decreases from 92.23 to 61.32%. which indicates that the pyrolysis temperature, and time determines the extent of the pyrolysis, and the yield of wax. At a low pyrolysis temperature (110˚C to 130˚C), from Figure-4 the yield of wax and reaction time has a low slope. When the temperature was increased to 150˚C, the yield decreases significantly and the slope increases as shown in Figure-4. There are two reasons for the decrease in yield, one is the pyrolysis of wax to more gaseous products as the temperature increases; the other reason is the vaporization of wax when pouring from the reactor into the mould. On the other hand, at very low temperature (110˚C to 120˚C) and short reaction time this is not feasible, because under this condition the polyethylene wax obtained showed some of the properties of the unpyrolysed waste sachets.

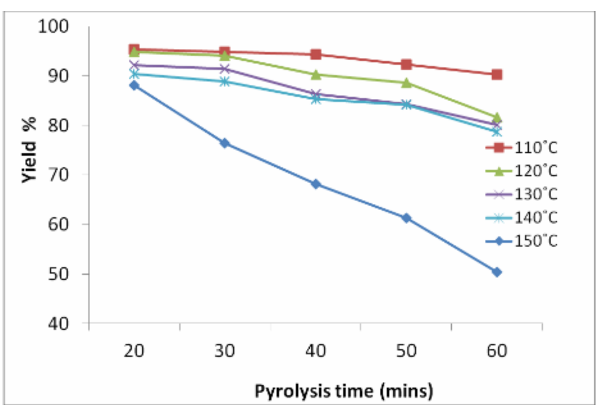

Figure-4. Yield of polyethylene wax obtained at different pyrolysis time and temperature.

Similar trend was observed by Jixing *et al.*, (2003) while pyrolysing waste plastics to produce polyethylene wax. Therefore 130˚C and 150˚C are the optimal temperatures and 20 minutes and 30 minutes are the optimal reaction times in other to produce wax with high yield i.e., over 90%. But these optimal temperatures and optimal reaction times will only be acceptable if penetration degree and the melting point of wax produced are within ASTM standard wax.

Effect of pyrolysis temperature and time on the penetration degree of polyethylene wax

The penetration degree of polyethylene wax at different pyrolysis temperature and pyrolysis time is shown in Table-1 and Figure-5. There was no change in the penetration degree for the first 30 minutes. Penetration degree is the index for the hardness of polyethylene wax; it is related to the molecular weight range and the pyrolysis deepness of polyethylene wax. The deeper the pyrolysis of polyethylene waste (pure water sachet), the lower the molecular weight range and the larger the penetration degree of polyethylene wax. It was observed that from Figure-5, when polyethylene waste sachets was pyrolysed at a temperature range from 110˚C to 150˚C, the penetration degree of Polyethylene wax obtained varies from 1 to 40.6mm. At certain pyrolysis temperatures, with an increase in pyrolysis time, the penetration degree of polyethylene wax obtained increases. At low temperatures, the penetration degree does not change significantly with an increase in temperature.

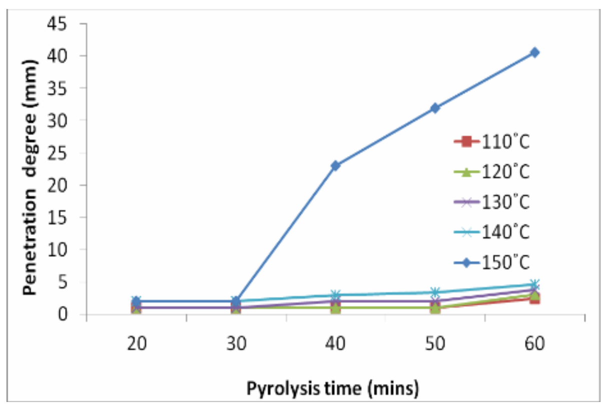

Figure-5. Penetration degree (0.1mm at 25^{o}C) at different temperature and pyrolysis time.

From the Figure-5 it was noted that there was a turning point in the curve which means that there exists a turning point in the reaction time (> 30mins) when temperature was 150˚C. At this temperature (150˚C), the penetration degree of polyethylene wax was over 40mm, which means that polyethylene waste will be over pyrolysed to wax as pyrolysis time increases and the yield will be too small. In order to produce wax with penetration degree higher than 10mm at lower temperature than 150˚C the prolysis time will be greater than 60minutes. Unlike polyethylene waste sachets wax with penetration degree of

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1 to 40.6mm obtained between 110˚C to 150˚C, Jixing *et al*., (2003) obtained a penetration degree of 0.1 to 42.4 mm while pyrolying waste plastics. Standard wax property requires that the penetration degree should be between 9 to 20mm (Wax properties, 2010) for paraffin wax and 14-45mm for microcrystalline wax; hence the wax produced from this waste sachet can be used to produce wax with both paraffinic and microcrystalline properties by carefully controlling the pyrolysis temperature and time within 130˚C to 150˚C.

The effect of pyrolysis temperature and time on the melting point of polyethylene wax

The melting point of wax at different pyrolysis temperature and time is shown in Figure-6 below. The melting point of polyethylene wax is determined by its molecular weight range and the extent of the pyrolysis of polyethylene. The deeper the pyrolysis, the lower the molecular weight range, the lower the melting point. Figure-6 shows that with an increase in pyrolysis time, the melting point of polyethylene wax decreases. It can be seen that if polyethylene waste water sachet was pyrolysed at the temperature range 120˚C to 150˚C, the melting point of polyethylene wax varies from 86˚C to 142˚C. At 110˚C the waste sachets were not fully pyrolysed to wax so the melting point was not determined at this temperature. At a temperature of 150˚C the melting point decreases sharply with a sudden decrease in slope after 30 minutes as shown in Figure-6.

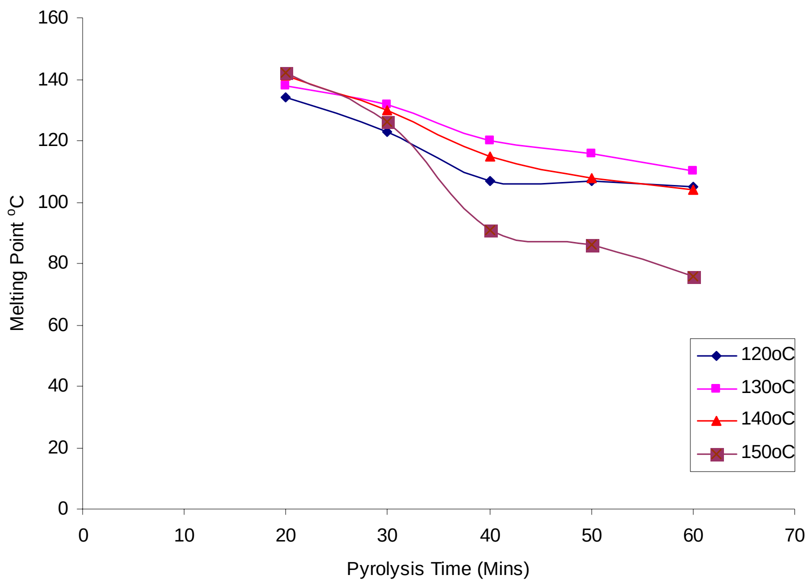

Figure-6. The melting point at different pyrolysis temperature and time.

In this study polyethylene wax with melting point of 142-76˚C was obtained at 110˚C to 150˚C waste from the waste sachets, Jixing *et al*., (2003) obtained a melting point of 144˚C-104˚C while pyrolying waste plastics. Standard wax property requires that the melting point of wax should be between 60-93˚C for microcrystalline wax (Wax properties, 2010), also industrial wax produced by Strahl and Witsch in Tables 1 and 2 showed a melting point of 122 - 202^{o}F (50 - 94^{o}C) for paraffin and microcrystalline wax, hence high quality wax which can be used for different purposes can be produced from this waste sachets pyrolysed at 130- 150˚C using well controlled pyrolysis time.

Gaseous products produced from the waste water sachets

The gas chromatogram of the non-condensable hydrocarbon gases produced along with the polyethylene wax between 50^{o}C-140^{o}C is presented Table-1. The analysis showed that only ethane 2.52%, propane 21.66% and propylene 75.82% were present. The result from Table-3 shows that during the production of wax from pyrolysis of pure water sachets these gases were produced. This implies that the gaseous products produced during the formation of wax from the sachets are very useful. The flame test of the gaseous product in Table-1, carried out during the pyrolysis of the sachets from between 50^{o}C-140^{o}C shows the gases produced (ethane, propane, propylene) burns with a blue flame.

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Table-3. Gas chromatography result of analyzed waste gaseous product.

Retention time (mins)	Components	Names	Concentration (mol/dm^3)	% Composition
2	C_2H_6	Ethane	0.0040	2.5
4	C_3H_8	Propane	0.0344	21.7
7	C_3H_6	Propylene	0.1205	75.8

The yield of hydrocarbon gases obtained at different pyrolysis time and temperature is shown in Figure-8. The yield of gaseous products obtained increased with pyrolysis time and temperature. The yield of gaseous products increased gradually at temperatures between 110°C and 140°C, but suddenly increased vigorously at 150°C. But in order to produce high quality wax with low melting point (50-93°C) and penetration degree of 9-60mm the water waste sachets must be pyrolysed below 150°C for 30-40 minutes to give wax yield of above 75% the remaining condensable hydrocarbon gases produced along with the wax which is mostly ethane, propane, propylene can be used as feedstock for the heater or sold as fuel gas.

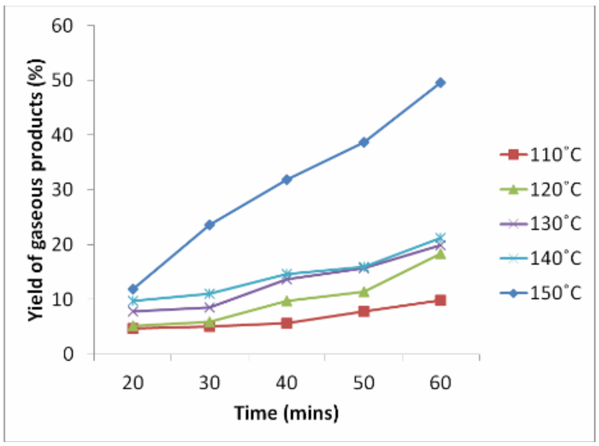

Figure-8. Yield of gaseous products obtained at different pyrolysis time and temperature.

CONCLUSIONS

Polyethylene waste was converted to polyethylene wax with a yield of over 90% optimum pyrolysis temperature of 130˚C-140˚C and optimum pyrolysis time of 20-30 minutes. The polyethylene wax obtained has a melting point of 76˚C-142˚C and penetration degree of the polyethylene wax obtained is 1-40.6 mm. Pyrolysis at high temperature say 150˚C and above results in low yield especially when pyrolysis time was increased. Waste sachets pyrolysed at 130-150˚C for 30-40 minutes produce high quality polyethylene wax (paraffin and microcrystalline wax) from waste polyethylene water sachets with yield of above 75%, good melting point and penetration degree in order to increase the yield the temperature at 150˚C and the reaction time should increased. The remaining condensable hydrocarbon gases produced along with the wax which is mostly ethane, propane, propylene can be used as feedstock for the heater or sold as fuel gas, hence waste water sachets can be used to produce microcrystalline polyethylene wax of high quality with great economic potentials. Work continues on the effect of temperature and time on the tensile strength, density of the wax produced from this waste sachet.

REFERENCES

Ademiluyi F. T. and Akpan C. 2007. Production of Fuel Oil from Pyrolysis of Waste Polyethylene (pure water sachets). Journal. Appl. Sci. Environ. Management. 11(3): 127-131.

Brebu. M., Bhaskar T., Murai K. and Muto. A. 2004. The Effect of PVC and / or PET on Thermal Degradation of Polymer Mixtures containing Brominated ABS. Fuel. 83(14): 2021-2028.

Jixing Shuyuan L., Xuan Wang and Xiangyang L. 2003. Study on the Conversion Technology of waste Polyethylene Plastic to Polyethylene Wax. Energy sources. 25: 77-83.

Strahl and Witsch. 2012. Paraffin and Microcrystalline Waxes Available: http://www.spwax.com/spparaff.htm {10th February, 2012}.

Owolabi R.U and Amosa M.K 2010. Laboratory Conversion of Used Water Sachet (Polyethylene) to Superwax Gloss Like Material. International Journal of Chemical Engineering and Applications. 1(1).

Wax properties. 2010. Available: http/www.igwax.com/wax properties.shtml. 31st January, 2012.

William R. Turner, Donald S. Brown and Donald V. Harrison. 1955. Properties of Paraffin Waxes Ind. Eng. Chem. 1933. 47(6): 1219-1226.