Original Article

Production and Blending of Sodium Based Water-Resistant Lubricating Greases from Petroleum and Petrochemical By–Products

Iheme C.[1], Offurum J.C.*[2] and Chukwuma F.O.[1]

[1]Department of Chemical Engineering, University of Port Harcourt, Nigeria
[2]Department of Chemical Engineering, Imo state polytechnic, Umuagwo-Ohaji

Address for Correspondence

Department of Chemical Engineering, Imo state polytechnic, Umuagwo-Ohaji
E-mail: jullyengine@yahoo.com

ABSTRACT

The present study is aimed at producing high water-resistant lubricating grease that can serve as a better alternative to the commonly used sodium-based grease. This is motivated by the need to reduce the high cost and upgrade the quality of Sodium-based greases available in our local markets motivated the present study. This is realizable by using polypropylene, low-density polyethylene, base-oils, and vegetable oils from Nigerian industries (petroleum and petrochemical industries). The quality of sodium – based greases obtained in the Nigerian market need to be improved. Most of these lubricants contribute to the problems encountered in ball-bearings of heavy-duty equipment, rather than the protection it is meant to provide. Moreover, these products are very expensive, and in most cases, they are imported from abroad. At the end, the grease produced from these by-products can compete effectively with the imported sodium and lithium base greases from abroad (USA) cost-wise. Measured quantities of polypropylene were melted separately in a measured quantity of base oil (500N and 650N/BS) by heating to a temperature of about 180^{o}C. Also sodium–base grease was prepared by a technology bounded by saponification, evaporation, melting of soap formed and blending with calculated quantity of base oil at the temperature ranging from 170^{o}C to 180^{o}C. Finally, the two products (melted polypropylene and sodium stearate in base oil) were blended at the temperature of about 100^{o}C. Quality control analysis was conducted on the product samples. The result obtained from the analysis showed that the product of the blends, when bright- stocked and used for blending 500N base oil, was within the ASTM and NLGI standard specifications for ball- bearing grease . The added value to the conventional sodium–base grease was that, the product can resist water up to 80 to 85%, unlike the conventional sodium grease in the present Nigerian market that mixes readily with water. However, the product of this research is economical in terms of cost and availability of raw materials in Nigeria, when compared with imported grease like Abro–products.

Keywords: Blending, Sodium base, Water-resistant, Lubricating Grease, Petrochemical.

INTRODUCTION

Grease is a semisolid lubricant. It generally consists of soap, emulsified with mineral or vegetable oil[1]. Greases are applied to mechanism that can only be lubricated infrequently, and where lubricating oil would not stay in position. They also act as sealant to prevent ingress of water and incompressible materials. Grease lubricated bearings have greater frictional characteristics due to their high viscosity[2]. American Society for Testing of Materials (ASTM D288 – 61, 1978) in petroleum terms defines lubricating greases as a ''Solid-to-semi fluid'' product of dispersion of thickening agent in a liquid lubricant[3]. Grease is used in application where liquid lubricants cannot provide the required protection. It is easy to apply and requires little maintenance. The main properties of grease are that, it stays in place, provides sealing action, and provides extra film thickness.

Grease consists of three main components: thickener, base oil, and additives. Most grease is identified by their thickeners. The most widely used thickeners are: sodium-soap, calcium-soap, lithium-soap, poly-urea, Aluminium complex, polymer and so on[4]. The need to lubricate various machineries used in manufacturing, automotive engineering and transportation industries has made the provision of various lubricating greases inevitable for their satisfactory end or smooth running. The overall importance of lubricant is to ensure that economic and design life of equipment, machineries and plants are achieved with maximum cost through: maintenance of oil film between moving parts so as to minimize the frictional effect of metal - to - metal contact; removal of dirt from engine parts, thus keeping them clean and efficient; absorbing and dissipating excessive heat generation, thus effecting cooling; and lubricating almost every moving part in the mechanical equipment. The elements of a machine that require lubricants are those in relative motion viz: Gears, Bearings, Slides, Ways, Piston/Cylinders, Cams/Cam followers, Flexible couplings, Pulley/Wire ropes etc. Advanced polymer additives for greases provide superior water wash-off resistance in application that is exposed to high levels of direct or indirect water spray. Grease treated with ***Lubrizol-2002*** additive reduces the spray-off rate to 14%, in comparison with nearly 100% removal for untreated grease and 96% for competitive products used at comparative treating rates. This additive reacts with the soap thickener and becomes an integral part of the soap structure. Cost efficiency is improved when the thickening efficiency is improved by reducing the amount of soap required to formulate greases[5]. Thus, worked penetration rating, as measured in ASTM D-217 test (in line with NLGI rating), can be achieved with lower levels of soap.

However, this project work shall focus on the production of sodium-based grease with about 80% water resistant or 20% water wash-off, which is not common in Nigerian auto parts market at the present, and can compete effectively with any other international sodium-based grease at various conditions.

MATERIALS & METHODS

Grease production involves a batch process which incorporates a technology bounded by saponification, evaporation, melting of thickened soap in a mineral fluid (Base Oil), recycling/cut back, milling or homogenizing and filtration of dispersed thickener in base oil; this is followed by addition of additives and modifiers to form grease[6].

Saponification and Evaporation Stage

Saponification is the alkali hydrolysis of fatty matters to form soap[7]. 103 grams of caustic soda solution (prepared by dilution to a concentration of 26%, with the specific gravity of 1.275 at room temperature of about 30^0C) was weighed into a plastic container. Then 206 grams of palm oil was weighed into the reactor in the ratio of (1:2) and was heated to a temperature of 60^0C (saponification temperature), followed by the gradual addition of already weighed caustic soda into the reactor. The mixture of the two reactants was stirred for a period of (5) minutes depending on the mass of the reactants (fatty matter plus caustic soda solution), to saponify and form soap. The soap formed in the process was later heated up to a temperature of about (120^0C to 130^0C) (at atmospheric pressure), in other to remove water (by evaporation). This is the temperature at complete saponification, and the soap obtained at this stage is very thick and in a solid state.

Grease Formation and Milling Operation

After saponification and evaporation at about 130^0C, the thickened soap formed is further heated to a temperature of about 170^0C to 180^0C to melt by adding little quantity of base oil so as to reduce friction or ease the stirring. At the above specified temperature, the thickened soap melts to a liquid state and becomes gummy or sticky on little exposure to air. At this temperature (180^0C), the molten soap was dispersed in base oil by gradual addition and stirring until the grease was formed. Here, reasonable quantity of base oil was added (900m/s for 650N, 800mls for 500N, and 500mls for 150N) in the respective production made and dispersed in each of these base oil samples.

(NOTE: The total volume of base oil to be added per batch depends on the texture of the product sample on cooling to room temperature: say 30^0C).

However, before allowing the grease to cool to room temperature, milling operation was performed using locally fabricated grinder at the temperature of 80^0C. Milling is a form of homogenization. This method helped in reducing the particle size of the undispersed thickener in the base oil.

Blending Operation

Two hundred grams (200g) of dispersed polypropylene product and 200grams of sodium grease from the above were blended together, at the temperature of 100^0C, by heating and stirring the two in a reactor for a period of 10 -15 minutes, based on the quantity of the two products to be blended. At the end of this period, the product of the mixture was allowed to stay for 24hours (one day) to cool. Later on, blending ratios of sodium/polypropylene greases were altered in the form (1:2), (2:3) and (1:3) respectively.

Process flow sheet for the production of sodium and polypropylene/ polyethylene (co-polymer) greases.

(See fig. 1, 2 and 3.)

QUALITY CONTROL ANALYSIS

The final product was tested or analyzed for quality. The tests conducted include those of the Dropping point and worked penetration of each of the samples produced.

Worked Penetration (ASTM D217, IP50)

Worked penetration is the penetration of a sample of lubricating grease after it has been brought to 25^0C (77^0F), and then subjected to 60 double strokes in a standard grease worker, and penetrated without delay[8].

Method

400grams of grease sample was cooled to 25^0C, and later transferred into the grease worker cup. The grease cup was Jared sharply on a bench to remove the air pockets, and the grease cup was finally filled above the rim of the cup and scraped at an angle of 45^0C (with the aid of spatula). The vent cock was closed and the grease inside the cup was subjected to 60% full double strokes of the plunger, completed in about one minute by timing it using a stop-watch; the plunger was finally returned to its top position at the end of the one minute.

The vent cock was opened; the top and plunger were then removed. The plunger itself was scraped with spatula to remove all the grease particles that cling to it. The seta cup was used to fill the worked grease sample in readiness for penetration test. After loading the cup with grease, the excess grease was scrapped off above the rim of the cup by moving the blade of the spatula, held inclined towards the direction of the motion at an angle of 45^0C across the rim of the cup.

PENETRATION MEASUREMENT

The test specimen was placed on the penetrometer table with one of the prepared faces upward. The mechanism was set to hold the cone in the "zero" position and the apparatus was adjusted such that the tip of the cone just touched the surface at the centre of the test sample. Finally, the cone shaft was released rapidly and was allowed to drop for five (5) seconds. The indicator shaft was gently depressed until it touched the cone shaft and penetration reading was taken from the indicator. The experiment was repeated up to three times, and the average result was recorded.

Dropping Point of Lubricating Grease (Astm D566, IP 132)

The ASTM–IP dropping point is the temperature at which a conventional soap–thickened–grease passes from a semi–solid to a liquid state under the conditions of test; this represents the temperature at which certain non–soap–thickened greases rapidly separate oil[9].

Method

The apparatus consist of small chromium-plated brass cup, a heat–resistant glass test tube with three indentations equally spaced on the circumference, a thermometer with ranges of -5 to 300^0C, a heating bath and an oil bath. Castor Oil fluid for an oil bath was used for the experiment. Grease sample was placed in the cup and a cone–shaped space created, by means of a rod manipulated through the orifice. A thermometer occupies the space out of contact with the grease. Heating took place at specified rate and the temperature at which oil falls or drops through the orifice of the cup to the bottom of the test tube was noted. The operation was repeated three times as a check (for consistency) of the result, and the average reading was determined and recorded.

RESULT AND DISCUSSION

The results of the analysis are presented in figures 4 – 6, as well as in tables 1 – 4. The data generated show that worked penetration increases with decrease in dropping point down the capacity (figures 4 and 5). This indicates that the higher the working capacity of the lubricant, the more the flow rate (that is the lesser the viscosity). Sample A had the highest dropping point (190^oC) when compared with samples B and C that had 180^oC and 185^oC respectively at 650N working capacity (Tables 1 – 3). This is traceable to the effect of the thickener in the high-density base oil (650N or Bright Stock), which is in agreement with similar reviews[10&11]. It could be, also, observed that the dispersion of the grease samples were highest at 100N working capacity, as indicated in figure 6. The results of physical analysis in tables 1 to 4, also, indicates that 10% of dispersed polypropylene in 650N (or bright stock) base oil is ideal to fortify sodium grease in the ratio of 1:1, in order to produce water resistance grease suitable for servicing of bearings and other heavy duty equipment.

However, the results obtained from the present study generally show that water-resistant-sodium-based grease and polyethylene (co-polymer) grease could be produced from petroleum and petrochemical by-products. Also a blend of normal sodium grease and melted polypropylene is ideal quality grease suitable for ball bearing based on ASTM/NLGI consistency grading.

CONCLUSION

The present study shows that suitable and ideal concentration of caustic soda, for soap manufacture, is 26% and it corresponds with the specific gravity of 1.275 at the temperature of 30^{o}C. The density of dispersed polyethylene/polypropylene in a fixed volume of base oil increases with increase in the mass of polyethylene/polypropylene dispersed. Also, 10% of dispersed polypropylene in 650N (Bright stock) base oil is ideal to fortify sodium grease in a good ratio (such as 2:1) in order to produce water resistance grease suitable for servicing bearings and other heavy duty equipment. Caustic Soda, fatty materials, high density base oils (650N / 500N), polypropylene and other additives are reliable raw materials for water resistant sodium base grease production, while low density polyethylene and polypropylene are recommended for water resistant light grease production. Sodium grease No. 3 is a ball-bearing and heavy duty greases, while polyethylene No. 2 grease is a multipurpose grease for general servicing of equipment or machines. It will serve as a substitute to imported lithium grease.

It is, also, recommendable that further work be done on the feasibility study of this project using co-polymer (polyethylene and polypropylene) as raw materials for production of light grease as a substitute for lithium grease.

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Table 1. Result of analysis of sodium/polypropylene grease in the Ratio of 1:1 (sample a)

Sodium/polypropylene = 1:1	Sodium plus polypropylene Grease with:			
Properties	650N	500N	150N	100N
Dropping point (^{0}c)	190	165	150	120
Worked penetration	230	260	320	340
Appearance	Smooth and fibrous	Smooth and fibrous	Buttery and fibrous	Buttery and fibrous
Other Properties	Adhesive and cohesive	Adhesive and cohesive	Adhesive and cohesive	Adhesive and cohesive

Table 2. Analysis of Sodium/Polypropylene Blended-Based Grease In The Ratio Of 1:2 (Sample A)

	Sodium plus polypropylene Grease			
PROPERTIES	650N	500N	150N	100N
Dropping point	180	170	140	130
Worked penetration	230	270	320	345
Water resistance	Fair to good	Fair to good	Fair to good	Fair to good
Appearance	Smooth & fibrous	Smooth & fibrous	Smooth & fibrous	Smooth & fibrous
Other properties	Adhesive & cohesive	Adhesive & cohesive	Adhesive & cohesive	Adhesive & cohesive

Table 3. Result of Analysis of Polyethlene Co-Polymer Grease (Sample B)

	Polyethylene Grease			
Properties	650N	500N	150N	100N
Dropping point (^{0}c)	185	180	150	120
Worked penetration	280	290	330	340
Appearance	Smooth and buttery	Smooth and buttery	Smooth and soft	Smooth and soft
Other Properties	Cohesive	Cohesive	Cohesive	Cohesive
Properties	650N	500N	150N	100N

Table 4. Worked penetration of samples at varying grades of base oils

Base oil grades	Sample A	Sample B	Sample C
650N	230	280	275
500N	260	290	290
150N	320	330	320
100N	340	340	340

Table 5. NLGI/ASTM Grade of Sodium-Based and Low-Density Polyethylene-Based Greases

NLGI COSISTENCY NUMBER	A.S.T.M PENETRATION VALUE (10^{th}/mm)	DROPPING POINT (^{o}C)	RESULT OBTAINED FOR SODIUM-BASED GREASE PRODUCED	RESULT OBTAINED FOR LOW-DENSITY POLYETHYLENE-BASED GREASE PRODUCED
2	265-295	160 minimum		270-280
3	220-250		230-240	

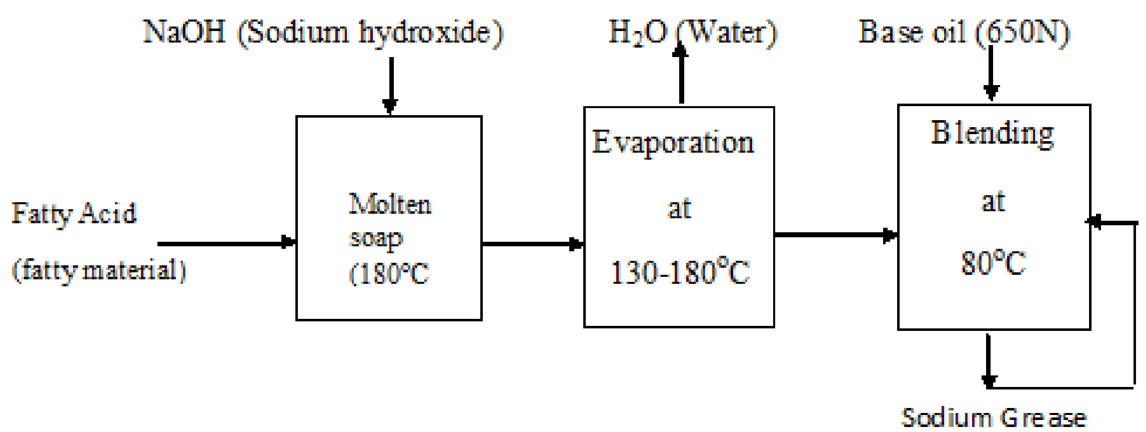

Figure 1. Sodium Base Grease Production (SAMPLE A)

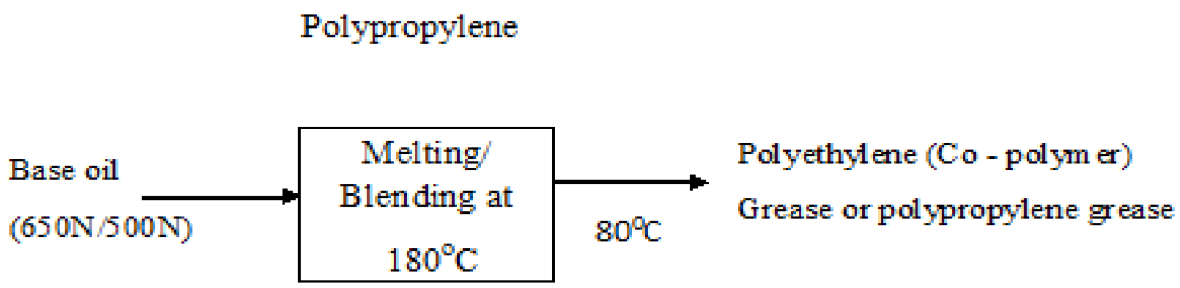

Figure 2. Co-polymer Grease Production (SAMPLE B)

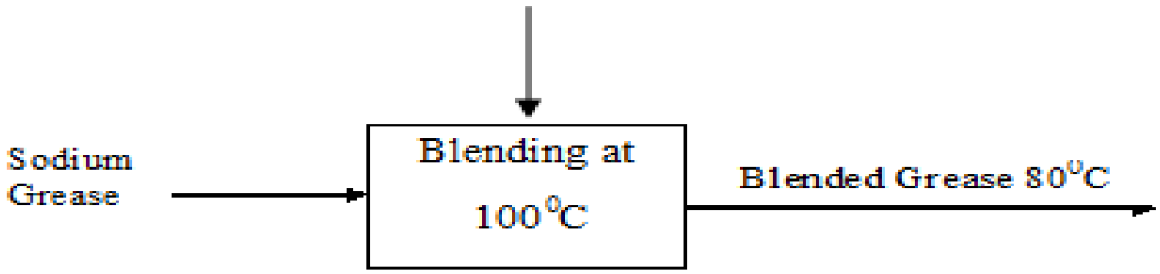

Figure 3. Blending of Sample A- *Sodium Base Grease* and Sample B- *Copolymer Grease*

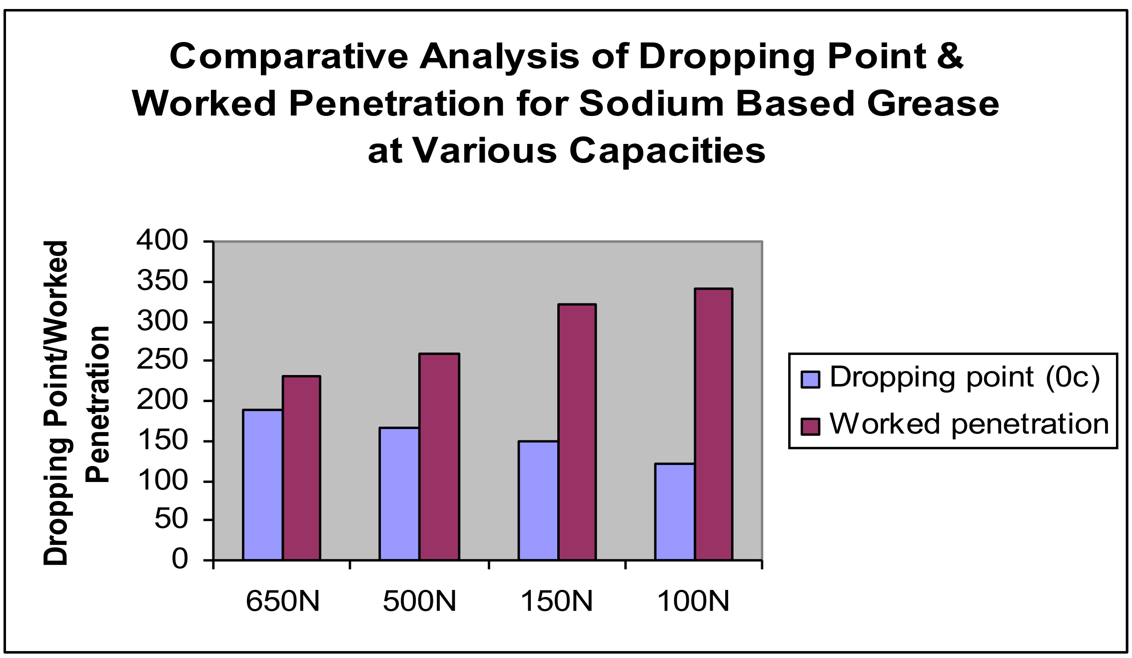

Figure 4. Comparative analysis Plot of dropping point versus worked penetration of sodium/polypropylene blended based grease in the ratio of 1:2

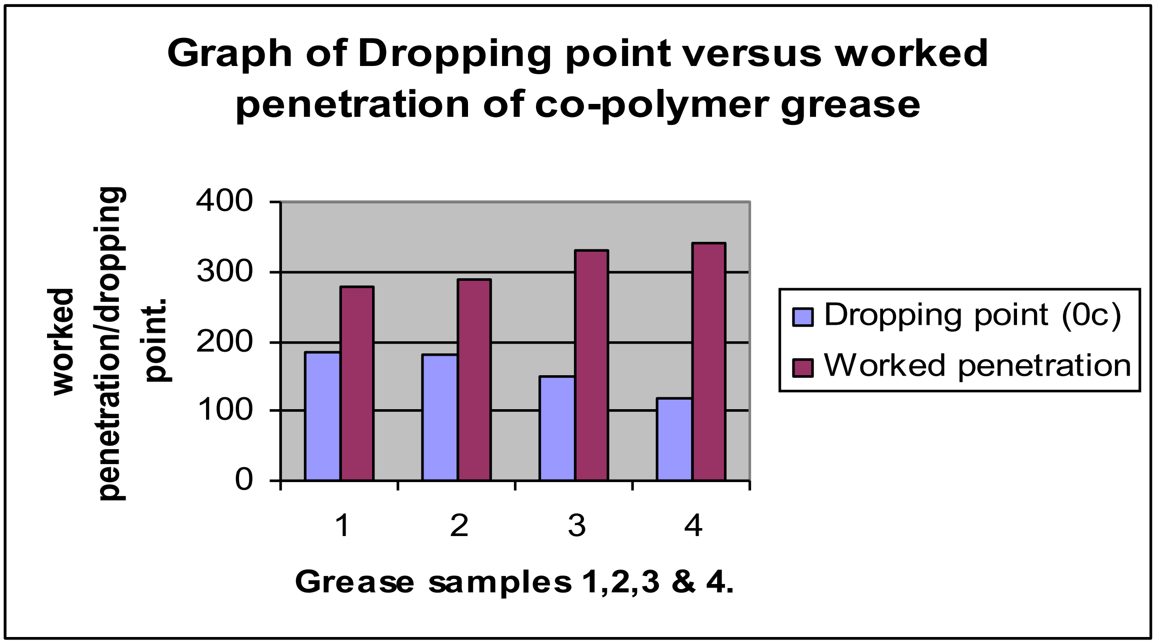

Figure 5. Graph of worked penetration (10^{th}/mm) versus dropping point(^{0}C) of polyethylene greases(where Sample 1=650N; 2=500N; 3=150N; 4=100N**)**

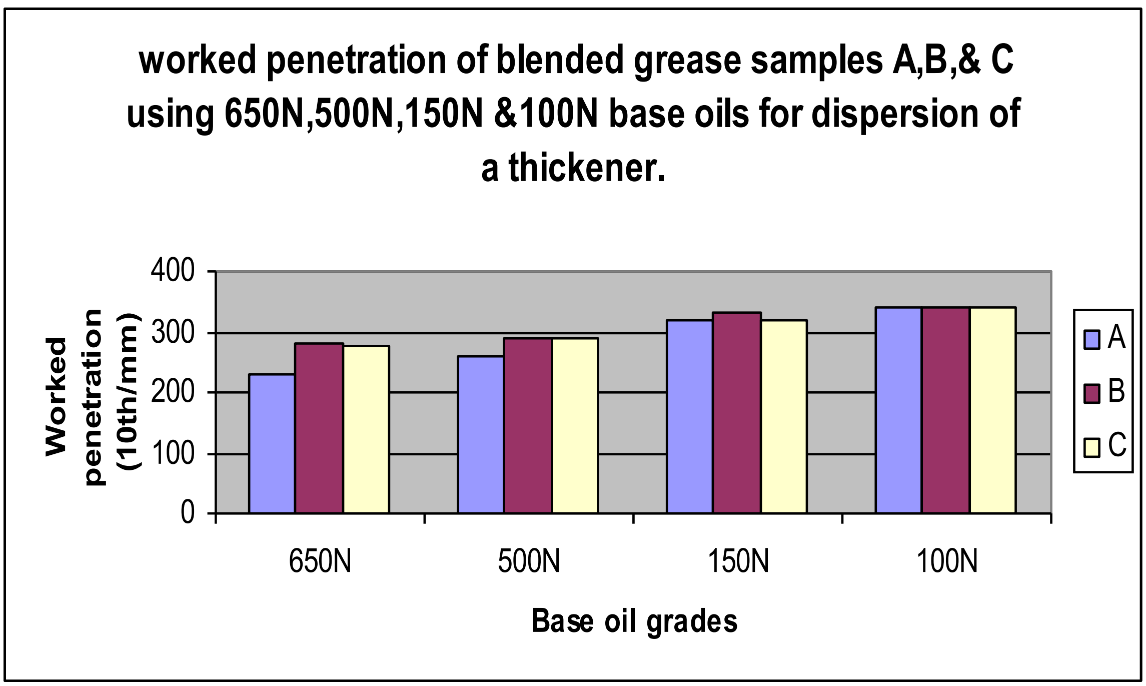

Figure 6. Worked penetration of samples at varying grades of base oils