World Journal of Engineering Research and Technology
WJERT
www.wjert.org
SJIF Impact Factor: 5.924

PHYSICOCHEMICAL PROPERTIES OF LITHIUM-BASED GREASE

[1]*Zafar Iqbal, [2]Zareen Ghafoor, [1]Muhammad Usman Sabri and [1]Mubeen Akhtar

[1]Applied Chemistry Research Centre, PCSIR Laboratories Complex, Lahore-54000-Pakistan.
[2]Department of Chemistry, Queen Mary College, Lahore.

Article Received on 06/10/2023 **Article Revised on 27/10/2023** **Article Accepted on 17/11/2023**

***Corresponding Author**
Zafar Iqbal
Applied Chemistry Research Chemistry Research Centre, PCSIR Laboratories Complex, Lahore.

ABSTRACT

Lithium-based grease was prepared by using stearic acid as thickener from auto oil 650. Concentrations 12, 15, 20 and 25% stearic acid was applied and grease was evaluated for cone penetration and drop point. Results revealed that cone penetration of grease was decreased 241, 239, 191 and 181 1/10 mm respectively for 12%, 15%, 20% and 25% stearic acid. While drop point increase 82 °C, 97 °C, 119 °C, and 125 °C respectively by increasing concentration of thickener from 12%, 15%, 20% and 25%. Furthermore, FTIR analysis was performed to observe the lithium bond, and carboxyl peaks, these peaks were clearly shown in all the FTIR spectra, which confirm the reaction of lithium hydroxide andcarboxylic acid.

KEYWORDS: Grease, lithium, thickener, stearic acid, cone penetration, drop point.

1. INTRODUCTION

Lubricating grease is a highly structured colloidal dispersion system made up of thickeners, additives, and lubricating base oil.[1] Typically, a grease composition is made up of 60–95% base fluid (mineral, synthetic, or vegetable oil), 5-25% thickening (fatty acid soaps of alkali or alkaline metals) with 0–10% additives (antioxidants, corrosion inhibitors, anti-wear/extreme pressure, antifoam, tackiness agents, etc.). The thickener, which is effectively the gelling ingredient, binds the matrix together while the base fluid gives the grease lubricating characteristics. When the remaining oil is added to the reaction mixture, the soap structure expands as a result of the base oil absorbing and adhering in the soap structure first.[2] There are two types of greases based on the type of thickener. Non-soap-based greases have the non-soap-based thickeners such as gelling agents instead of metallic soaps. It has an important characteristic of being insoluble in liquid phase because the gelling agent is insoluble in liquid phase at all temperature. Non soap thickeners used includes Inorganics i.e., carbon black, silica and organophilic clays. Organics i.e., sodium octadecyl terephthalate, aryl-substituted ureas, copper phthalocyanines and indanthrene blue.[3][4] Soap-based greases have soap-based thickeners such as metallic soap. It might be any metal soap grease, including calcium soap, lithium soap, aluminum soap, and others. One of the first known greases, calcium soap grease, is both mechanically stable and water resistant. The normal dropping point for calcium soap grease is 95 °C. The structure of sodium soap grease is fibrous, and it can withstand relatively high temperatures but not water. Compared to calcium grease, sodium soap grease has a higher dropping point (175 °C). Although lithium-based grease often seems smooth, there might be a grain structure. Lithium soap grease combines the high-temperature qualities of sodium soap grease with the water resistance of calcium soap grease.[5] The National Lubricating Grease Institute's (NLGI) technique of classifying grease based on consistency class determined by penetration value is widely utilized internationally.

Table 1.1: NLGI grease classification.[6]

NLGI number	Penetration (0.1mm) at 25 C	Consistency
000	445-475	Semi fluid
00	400-430	Semi fluid
0	355-385	Very soft
1	310-340	Soft
2	265-295	Common grease
3	220-250	Semi hard
4	175-205	Hard
5	130-160	Very hard
6	85-115	Solid

2. MATERIALS

2.1 Chemicals and equipment

The material required for experiment was Stearic acid (Molecular weight=284.49g/mol, Melting point=67-70 °C, White flaky crystal with luster), Lithium hydroxide Molecular weight=23.95g/mol, Melting point= 462 °C, Density= 1.46 g/cm3, Boiling point= 924 °C), Engine oil SAE-20W-50 (auto oil 650) and Methanol (Molecular weight=32.04g/mol, Melting point=-97.6 °C, Boiling point=64.7°C, Density =0.79g/mol). The equipment required were Hot plate, cone penetrometer and FTIR spectrophotometer.

3. METHODOLOGY

3.1 Preparation of grease

In a beaker, 3g of LiOH was weighed and mixed with 10ml of water. The solution was heated until all of the LiOH had dissolved completely. In the steel jar, 70g of Auto oil 650 was poured and heated to 80°C. After that, turn by turn 12g, 15g, 20g, and 25g of stearic acid was added to the oil and continually swirled. With continual stirring, LiOH solution was added to the stearic acid and oil mixture. The entire mixture was heated to 110 °C for nearly an hour. The grease had begun to develop. The temperature of the grease was lowered to around 50 °C and 2 milliliters of 99 percent methanol were added to give the grease its final structure.[7]

3.2 Drop point test D566

The temperature at which grease transitions from a semi-solid to a liquid form under test circumstances is known as the dropping point. The ASTM D-566 procedure was utilized.[8] The dropping point test was used to measure the cohesiveness of an oil and the thickener of a grease. For quality control, the dropping point test was performed to ensure that the soap structure was formed appropriately and could survive high temperatures. A thermometer was attached to a cork, and the tip of the thermometer was covered in the grease sample to be tested. The cork was then inserted into the flask's mouth. The entire setup was placed on top of a heating device (hot plate). The temperature of the hot plate was gradually increased. The grease softened as the temperature was raised. The grease was totally melted and dropped at a specific temperature. Temperatures that corresponded to the melting point were recorded.[9]

3.3 Cone penetration test D-217

This test measured the consistency of lubricating grease by penetration of the standard cone. ASTM D-217 method was used. A penetrometer shall be capable of indicating depth in tenths of a millimeter, since cone shaft rapidly released, and allowed to drop for 5.0 sec. The penetration was read and recorded from the indicator. The cone penetration test was performed to assess the greases' consistency. The cone penetration value was also used to determine NLGI grades and mechanical stability of the greases. In order to perform the cone penetration test, the grease was poured into the worker's cup and smoothed on the surface. The greased was heated to 25 °C. The penetrometer was set so that the pointer was on the scale's zero position. The tip of the penetrometer's cone was adjusted so that it just touched the grease's surface. The penetrometer's cone assembly was then released, and the cone was allowed to freely fall into the grease for five seconds. The scale was used to record the reading.[10]

3.4 FTIR analysis

Lithium grease made with 12%, 15%, 20%, and 25% of stearic acid underwent FTIR examination and following peaks were observed.[11]

3.4.1 FTIR analysis of lithium-based grease prepared with 12% of stearic acid

The FTIR spectrum of grease prepared from 12g of stearic acid had five peaks which corresponded to specific wave numbers indicating the specific functional groups.

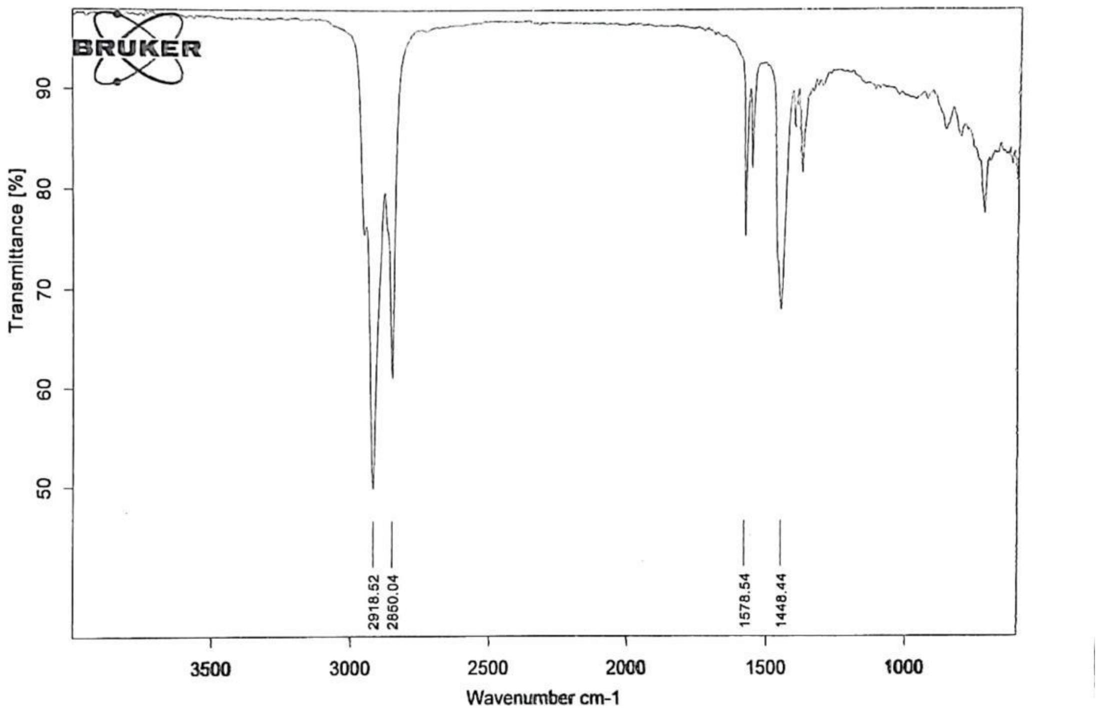

Figure 3.1: FTIR spectrum of lithium-based grease prepared with 12 g of stearic acid.

The vibration at 2918.52cm^{-1} indicated the stretching of the C-H bond in the methyl group. The peak at 2850.04cm^{-1} confirmed the presence of the methylene group as it corresponds to the C-H sym. Stretching of methylene. The peak at 1578.54cm^{-1} indicated the presence of carboxylic acid salt, and the peak at 1448.44cm^{-1} may be either due to C-H bending of methylene or asym. bending of the methyl group. No absorption peak in the range of 3000-3500cm^{-1} indicated that all the OH groups were utilized in the formation of soap and the peak at 710 to 720cm^{-1} indicated the presence of O-LiOs stretching.

3.4.2 FTIR analysis of lithium-based grease prepared with 15% of stearic acid

The grease prepared from 15g of stearic acid was subjected to FTIR spectroscopy. Six different bands were observed which represent different functional groups based on the vibrations of the bonded atoms.

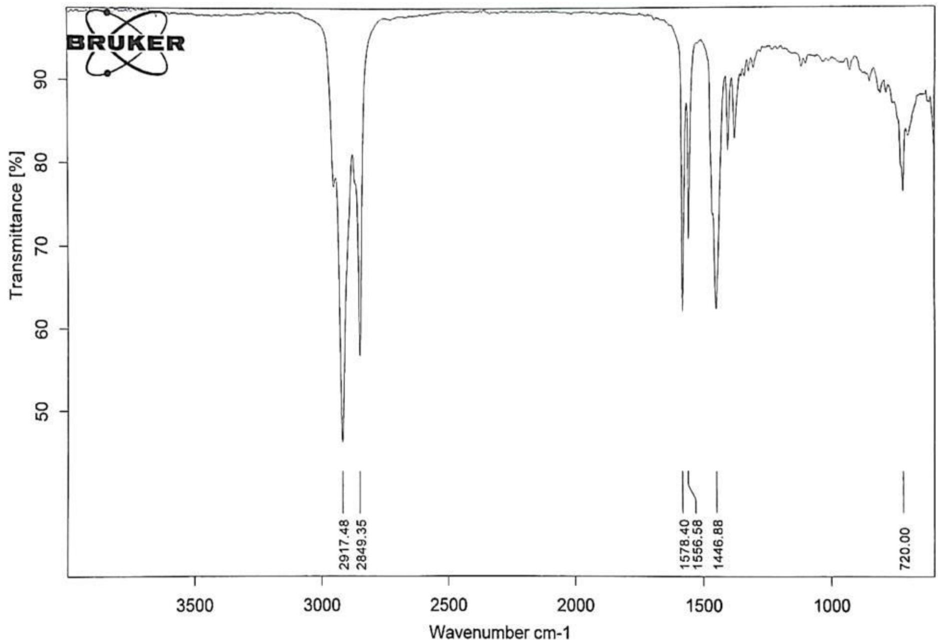

Figure 3.2: FTIR spectrum of lithium-based grease prepared with 15g of stearic acid.

At 2917.48cm-1, stretching due to the C-H bond of methyl was observed. The 2849.35cm-1 peak was due to the C-H acid salt. Each of the recorded peaks corresponded to a specific functional group, as the peak at 720cm^{-} sym. stretching of methylene. The wave number or frequency at 1578.40cm^{-1} and 1556.58cm^{-1} indicated the presence of carboxylic [1] confirmed the OLiOs stretching vibrations. Because all –OH bonds were used in the formation of soap, there was no peak in the region 3000-3500cm-^{1}. Peak at 1446.88cm^{-1} was either due to C-H bending of methylene or C-H asym. Bending of the methyl group and the peak at 710 to 720cm^{-1} indicated the presence of O-LiOs stretching.

3.4.3 FTIR analysis of grease prepared with 20% of stearic acid

The grease prepared from 20g of stearic acid was subjected to FTIR spectroscopy. Different peaks were observed due to different functional groups.

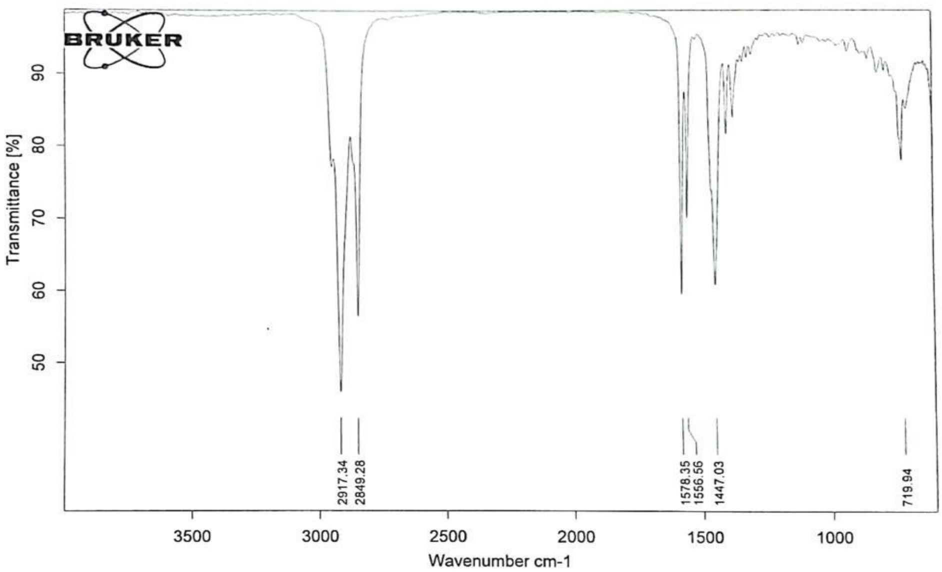

Figure 3.3 FTIR spectrum of lithium-based grease prepared with 20g of stearic acid.

Methyl C-H stretching vibrations and methylene C-H asymmetric were observed at 2917.34cm-[1]. Stretching was observed at 2849.28cm^{-1}. Two peaks were close together at 1556.56cm^{-1} and 1578.35 cm^{-1} indicating the presence of carboxylic acid salt. The stretching vibrations of the OLiOs bond occurred at 719.49cm^{-1}. Because all –OH bonds were used in the preparation of soap, no peak was observed at 3000-3500cm^{-1}.

3.4.4 The FTIR spectroscopy spectra of grease prepared from 25% of stearic acid

showed different peaks at different wave numbers, indicating the presence of specific functional groups depending on the frequency of vibration.

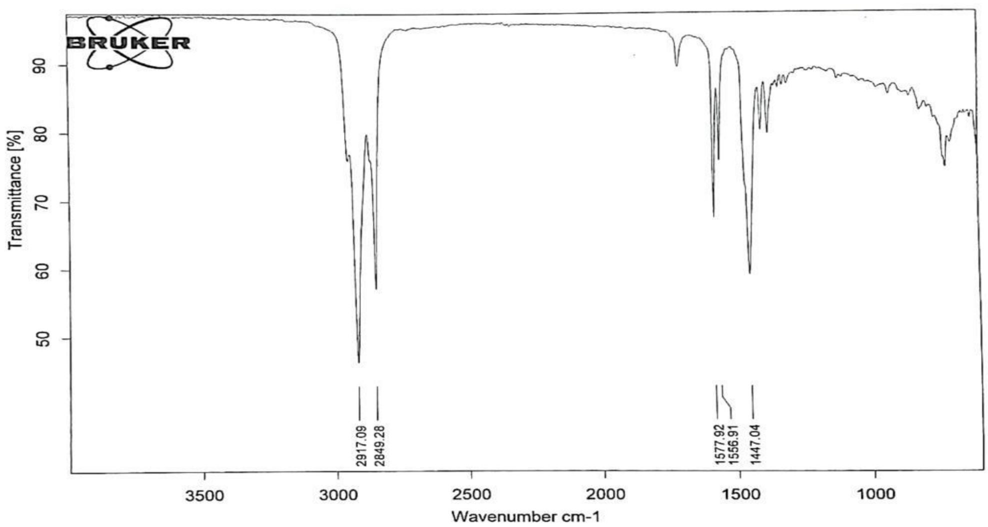

Figure 3.4 FTIR spectrum of lithium-based grease prepared with 25g of stearic acid.

The stretching vibrations at 2917.09cm^{-1} and 2849.28cm^{-1} correspond to the C-H stretching of Methyl and C-H asym. Methylene stretching, respectively. The peak at 1577.92cm^{-1} and 1556.91cm^{-1} was due to vibrations of the carboxylic acid salt group. Peaks were not observed in the region 3000-3500cm^{-1} because almost all of the OH groups were used in the formation of soap. The peak at 1447.04cm^{-1} was either due to methylene C-H bending or methyl C-H asym. Bending and the peak at 710 to 720cm^{-1} indicated the presence of O-LiOs stretching.

4. RESULTS AND DISCUSSIONS

The cone penetration and drop point of the grease were evaluated. NLGI grades were assigned according to cone penetration values. The appearance of greases was observed given in table 4.1.

Table 4.1: Appearance, cone penetration, drop point and NLGI grades of prepared grease.

Stearic acid concentration	Appearance	Cone penetration 1/10 mm	Drop point	NLGI grade
12%	Brown color	241	82 °C	3
15%	Dark brown	239	97 °C	3
20%	Dark brown	191	119 °C	4
25%	Dark brown	181	125 °C	4

4.1 Comparison of Lithium based greases cone penetration with stearic acid concentration

The cone penetration was affected by varying the quantity of stearic acid. Various types (a, b, c, d) of lithium-based greases were prepared with 12g, 15g, 20g and 25g of stearic acid and their cone penetration values were compared. The values of cone penetration reduced from 241 to 181. The best lithium-based grease was type d grease which was prepared with 25g of stearic acid and had a cone penetration value 181. It was observed that with the increase in concentration of the stearic acid, the cone penetration of the grease decreased. With the increase in concentration of stearic acid, the thickness of the grease increased, and because of this increased thickness, penetration of the cone decreased. Grease prepared from 12g of stearic acid had cone penetration 241. Grease prepared from 15g had a lower cone penetration value of 239. In the same manner, grease prepared from 20g of stearic acid had a cone penetration value of 191. Grease prepared from 25g of stearic acid had a 181-cone penetration value. With the increase in concentration of stearic acid from 12 to 25 g, the cone penetration value decreased from 241 to 181. The NLGI number of greases prepared from

12g and 15g had NLGI grade3, and the grease that used 20g and 25g of stearic acid was 4.

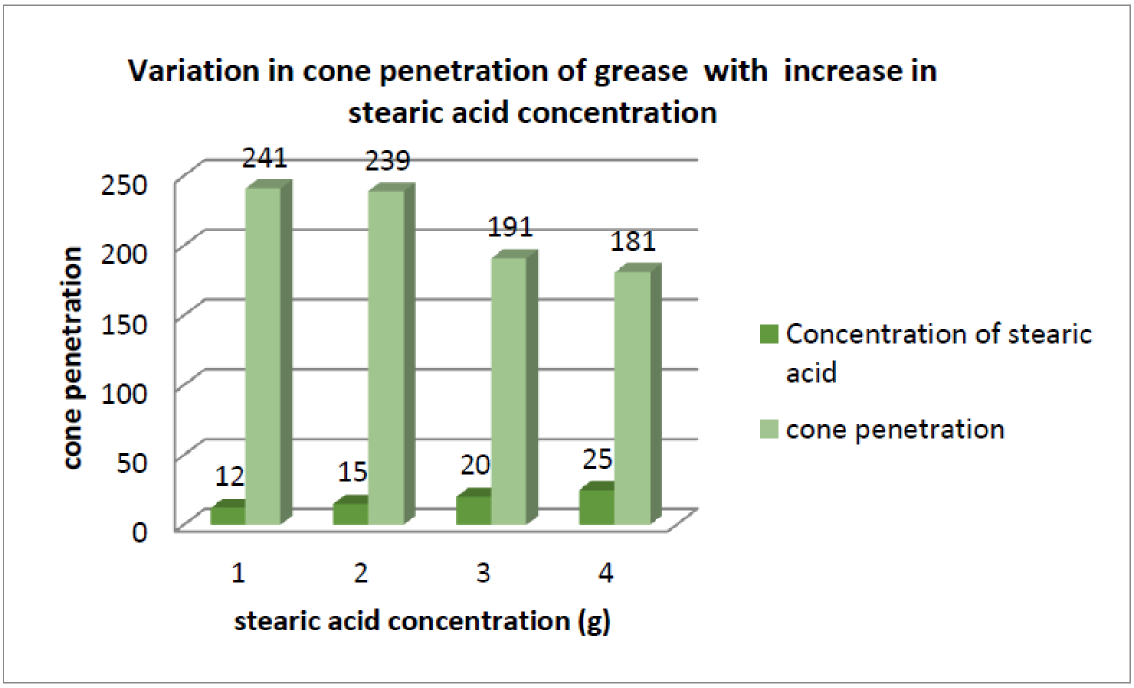

Figure 4.1: Graph presenting the variation of cone penetration with increased concentration of stearicacid(g).

4.2 Comparison of lithium-based greases dropping point with stearic acid concentration

By altering the percentage of stearic acid, four distinct types of lithium-based grease (a, b, c, d) were created, and changes in grease consistency affected the dropping point. Because of the change in the consistency of the grease, the dropping point rose from 82°C to 125°C. The grease type d made from 25g of stearic acid was the best since it had great temperature resistance and was used in machinery components that needed to be worked at high temperatures**.** The melting point of the grease increased as the concentration of stearic acid increased. The thickness of the grease increased as the concentrationof stearic acid increased. So, it took more time to get melted and drop from the thermometer, so the dropping point was also increased. The grease was melted at a higher temperature with an increase in the concentration of stearic acid. The dropping point of the grease containing 12g, 15g, 20g, and 25g ofstearic acid was found to be 82 °C, 97 °C, 119 °C, and 125 °C respectively. From the results, it was found that the concentration of stearic acid had a direct relation with the dropping point, so an increasedorder of dropping point was observed.

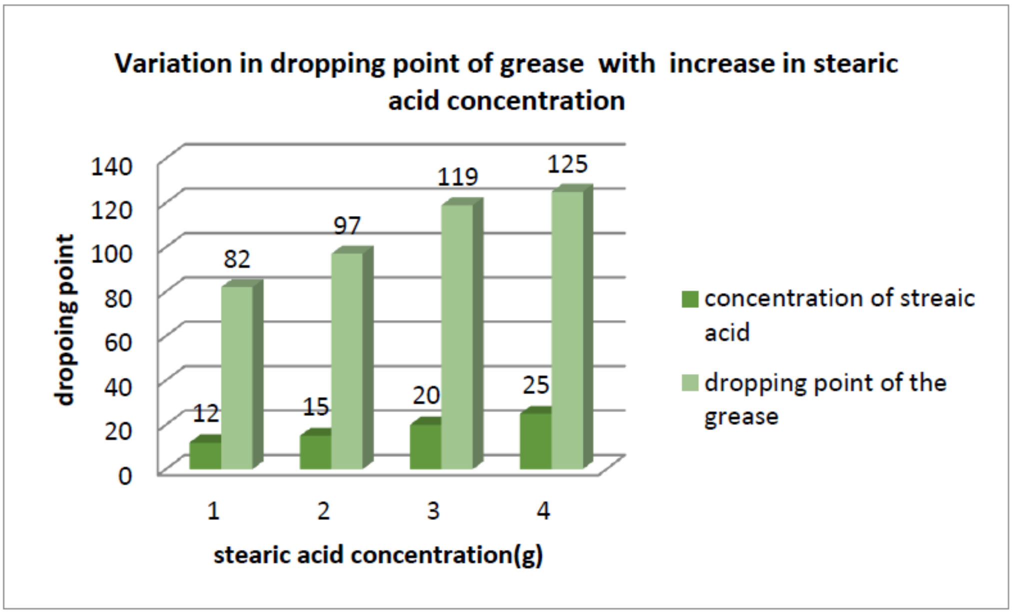

Figure 4.2 Graph presenting the variation of dropping point of grease with increased concentration ofstearic acid.

5. CONCLUSION

Lithium-based grease was prepared by using stearic acid as thickener from auto oil 650. Concentrations12, 15, 20 and 25% stearic acid was applied and grease was evaluated for cone penetration and drop point. Results revealed that cone penetration of grease was decreased 241, 239, 191 and 181 1/10 mm respectively for 12%, 15%, 20% and 25% stearic acid. While drop point increase 82 °C, 97 °C, 119 °C, and 125 °C respectively by increasing concentration of thickener from 12%, 15%, 20% and 25%. Furthermore, FTIR analysis was performed to observe the lithium bond, and carboxyl peaks, these peaks were clearly shown in all the FTIR spectra, which confirm the reaction of lithium hydroxide andcarboxylic acid.

REFERENCES

1. Chen, C., Liu, Y., Tang, Q., Xu, H., Tang, M., Li, X., ... & Dong, J. (2023). Tribological andrheological performance of lithium grease with poly-α-olefin and alkyl-tetralin as base oils. *Chinese Journal of Chemical Engineering*, *56*: 180-192.
2. Adhvaryu, Atanu; Erhan, Sevim Z.; Perez, Joseph M. (2004). *Preparation of Soybean Oil-Based Greases: Effect of Composition and Structure on Physical Properties. Journal of Agricultural and Food Chemistry, 52(21): 6456–6459.*
3. Saatchi, A., Shiller, P. J., Eghtesadi, S. A., Liu, T., & Doll, G. L. (2017). A fundamental study of oil release mechanism in soap and non-soap thickened greases. *Tribology International*, *110:* 333-340.

4. Young, G. J., & Chessick, J. J. (1958). Basic factors in the formation and stability of nonsoap lubricating greases: I. A study of the factors responsible for gel formation. *Journal of Colloid Science*, *13*(4): 358-371.
5. . Mohammed, M. A. (2013). Effect of additives on the properties of different types of greases. *Iraqi Journal of Chemical and Petroleum Engineering*, *14*(3): 11-21.
6. Buhlak, S., Ibrahim, B., & Alhamoui, M. (2014). Manufacturing (lithium-sodium) lubricant grease based on Syrian base oil and studying its physical, chemical, and rheological properties. *Int. J. ChemTech Res*, *6:* 2247-54.
7. Siyuan, C., Xiaochuan, G., Jing, W., Yan, H., & Wanjie, L. (2018). Preparation and Characterization of Lithium Grease Using 9, 10-dioxystearic Acid. *Chemical Engineering Transactions*, *65:* 577-582.
8. Mohammed, M. A. (2013). Effect of additives on the properties of different typesof greases. *Iraqi Journal of Chemical and Petroleum Engineering*, *14*(3): 11-21.
9. Mohamed, A., Osman, T. A., Khattab, A., & Zaki, M. (2013). Influence of nano grease composite on rheological behaviour. *Int. J. Eng. Res. Appl*, *3*(6): 1126-1131.
10 Martín-Alfonso, J. E., Valencia, C., Sánchez, M. C., Franco, J. M., & Gallegos, C. (2011). Evaluation of different polyolefins as rheology modifier additives in lubricating grease formulations. *Materials Chemistry and Physics*, *128*(3): 530-538.
11 Nandiyanto, A. B. D., Oktiani, R., & Ragadhita, R. (2019). How to read and interpret FTIR spectroscope of organic material. *Indonesian Journal of Science and Technology*, *4*(1): 97-118.