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INSTITUTE OF NATURAL AND APPLIED SCIENCES

MSc THESIS

Ayşen YILMAZ

INVESTIGATION OF NEW OIL ADDITIVES

DEPARTMENT OF MECHANICAL ENGINEERING

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INSTITUTE OF NATURAL AND APPLIED SCIENCES

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We certify that the thesis titled above was reviewed and approved for the award of degree of the Master of Science by the board of jury on …/09/2010.

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ABSTRACT

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While the lubricants industry has been focused over the last century on development and production of lubricating oils and greases using mineral oils as the base, a number of factors have been considered for the development of products derived from renewable harvestable resources such as vegetable oils and esters produced from them.

In this work, the effects of new bio-based oil additives on mineral base oil quality were evaluated; in addition improvement of the conventional oil properties by blending with bio-based oil (Jojoba) was investigated. A Timken Test Device was designed and manufactured according to ASTM D2782 in order to determine out variation of the oil blends' viscosity values. The viscosity values of oil blends were determined with and without jojoba addition and they were compared with each other.

Key Words: Mineral Based Oils, Jojoba Oil, Timken Test Device, Lubricant Properties

ÖZ

YÜKSEK LİSANS TEZİ

YENİ YAĞ KATKILARININ ARAŞTIRILMASI

Ayşen YILMAZ

ÇUKUROVA ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ

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Son yüz yılda yağ endüstrisi mineral yağların baz olarak kullanılıp yağlayıcıların ve greslerin geliştirilmesi ve üretiminde yoğunlaşsa da, bitkisel yağ ve bunlardan elde edilen esterler gibi yenilenebilir ve tarımsal kaynaklardan elde edilen ürünlerin geliştirilmesi için de birçok faktör göz önünde bulundurulmuştur.

Bu çalışmada, yeni biyo tabanlı yağ katkılarının mineral baz yağının kalitesine etkileri değerlendirilmiştir ve buna ilaveten geleneksel yağ özelliklerinin biyo tabanlı yağların (Jojoba) karıştırılması ile iyileştirilmesi araştırılmış. Yağ karışımlarının viskozite değerlerindeki değişiminin belirlenmesi amacıyla ASTMD82 standardına göre Timken Test Cihazı tasarlanmış ve üretilmiştir. Jojoba yağı katkılı ve katkısız yağ karışımlarının viskozite değerleri belirlenmiş ve birbirleriyle kıyaslanmıştır.

Anahtar Kelimeler: Mineral Baz Yağlar, Jojoba Yağı, Timken Test Cihazı, Yağlayıcı Özellikleri

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LIST OF ABBREVIATIONS AND NOMENCLATURE

JO	: Jojoba Oil
SO	: Spindle Oil
LN	: Light Neutral
HN	: Heavy Neutral
BS	: Bright Stock
EP	: Extreme Pressure
VI	: Viscosity Index
PP	: Pour Point
CP	: Cloud Point
API	: American Petroleum Institute
P	: Poise
cP	: Centipoise
R_C	: Rockwell C hardness value

1. INTRODUCTION

All surfaces are rough. The world of the engineer is made of solids whose surfaces acquire their texture as the result of a great variety of processes. In some cases it is merely a byproduct of forming the bulk shape, for example, in casting, molding, or cutting. More often a separate process affecting only the surface layers is applied after the part has been formed to its bulk dimensions. Some treatments remove material, as in grinding and etching. Others, such as plating, flame spraying, and sputtering, add it. Yet others merely redistribute the surface layer: peening and calendaring are examples. In addition, surfaces often show the marks of unplanned treatments such as wear and corrosion **(**Williamson, 1988).

Almost all modern machines require the use of a lubricant. Power generation in such equipment is achieved by the use of engines that mostly comprise metal parts that move against one another. In many cases, there is metal to metal contact that leads to the generation of friction and heat, which results in wear. The extent of wear in equipment depends upon the degree of the metal to metal contact, either due to the equipment design or the nature of the operation. For example, the equipment that is designed to experience minimal metal to metal contact, as is the case in most parts of an internal combustion engine, there is little friction and wear. However, the parts that are designed to have intimate metal to metal contact, such as gears and bearings, wear due to friction is extensive. With respect to the effect of equipment operation on wear, high speed, low load operation leads to lower wear than slow speed, high load operation. This is because in the former case there is minimal metal to metal contact (Rizvi, 2009).

Lubricants today are classified into two major groups: automotive lubricants and industrial lubricants. Industrial lubricants can be sub-divided into industrial oils and industrial specialties. Specialties in this case are principally greases, metalworking lubricants and solid lubricant films (Mang and Dresel, 2007).

A lubricant can be a solid, liquid, or gas, and lubrication is its primary function. The usual objective of the lubrication is to lubricate surfaces to minimize direct metal to metal contact and, hence, reduce friction and wear. The term lubricant

is also loosely applied to many other fluids that do not specifically perform this function. Examples include power and heat transmission fluids, hydraulic fluids, dielectric fluids, process oils, and the others. A lubricant performs many diverse functions, which help protect and prolong the life of the equipment. These include the following:

- Lubrication (reduce friction and wear): Lubricant helps reduce friction and wear by introducing a lubricating film between mechanical moving parts, such as gears and bearings. Essentially, the presence of a lubricating film minimizes the metal-to-metal contact and reduces the force necessary to move one surface against the other, thereby reducing wear and saving energy.
- Cooling (heat transfer): Lubricant acts as a heat sink and dissipates the heat away from the critical moving parts of the equipment, thereby decreasing the possibility of the machine component deformation and wear. The heat is either frictional heat that results from the metal surfaces rubbing against one another, such as in gears, or is conducted and radiated heat, which is due to the close proximity of the parts to a combustion source, such as the combustion chamber in an automobile engine.
- Cleaning and Suspending: Lubricant facilitates smooth operation of the equipment by removing and suspending potentially harmful products, such as carbon, sludge, and varnish, and the other materials, such as dirt and wear debris. This lubricant function is important in operations that involve high operating temperatures, as in the case of an internal combustion engine or a transmission. This is because in these applications the lubricant gets oxidized to form deposit precursors that can separate on hot surfaces and get converted into deposits.
- Protection: Lubricant prevents metal damage due to oxidation products, corrosion, and wear. It achieves this by forming a physical film on metal surfaces that is impervious to oxygen, water, and acids, or by forming physical and chemical films by additives, such as rust and corrosion inhibitors, extreme pressure (EP) additives, and anti-wear agents, that are present in the lubricant.
- Transfer Power: Lubricant is used as a power transfer medium in some applications, for example, in hydraulic systems. The lubricant performs this function

in addition to its normal function of lubrication. Examples of equipment that use hydraulics technology include transmissions, circulating systems, lifts used in automotive service stations, log splitters, fork lifts, dump trucks, and underground continuous mining equipment such as drills, loaders, and miners. The world lubricant market for the year 2006 is estimated at around 38.5 million metric tons, with the United States consuming about 9.5 million metric tons, or almost 25 % of the total world use. Asia Pacific and the Near/Middle East have the next highest share and are the regions with the fastest growth in lubricant consumption. They are followed by Central and Eastern Europe, Western Europe, and the others. The 2005 world demand breakdown by geographic areas is presented in Table 1.1 and in Figure 1.1 (Rizvi, 2009).

Table 1.1. 2005 world lubricant consumption by region in million metric tons (Rizvi, 2009).

Region	**Estimated Use (mmt)**
North America	9.48
Asia Pacific, Near/Middle East, Africa	14.02
Central and Eastern Europe	4.80
Russia	1.67
Western Europe	4.69
Latin America	3.00
Others	0.24
Total	37.9

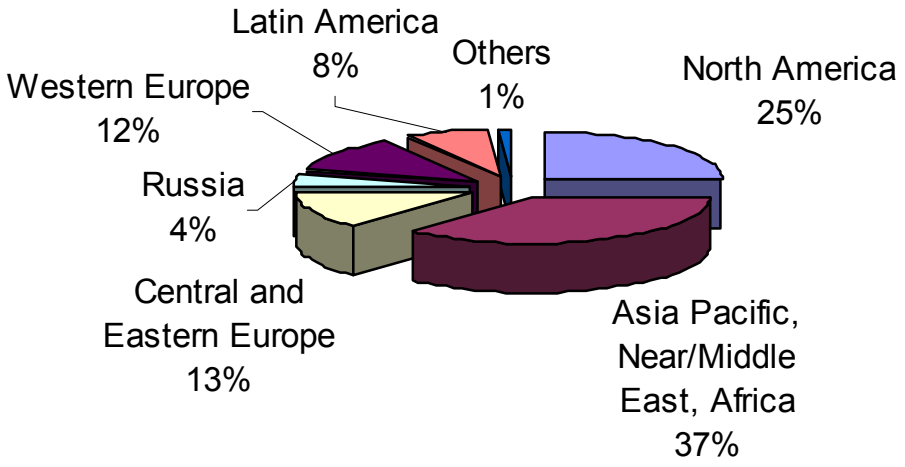


Figure 1.1. 2005 world lubricant consumption by region (Rizvi, 2009).

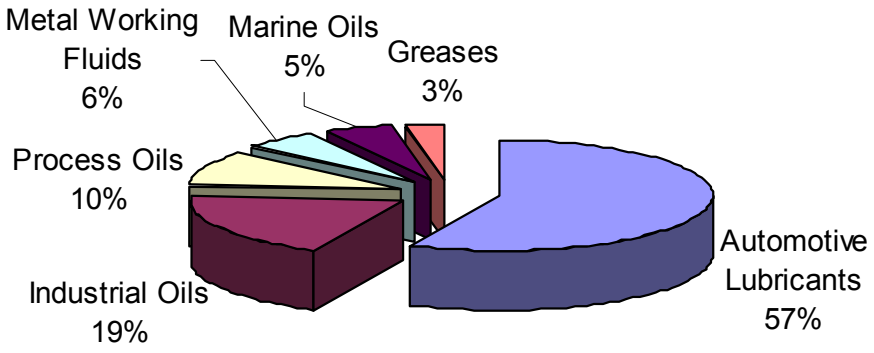


Figure 1.2. World lubricant use by product type (Rizvi, 2009).

The demand for lubricants is projected to increase at an average rate of 2% over the next decade, bringing the world lubricant consumption to 41.8 million tons in the next year. This estimate takes into consideration the expected growth rates of 5% for China, 3.5% for India, and 2.5 to 3.3% for Malaysia, Indonesia, and Thailand. Engine oils, that are the internal combustion engine lubricants, accounted for approximately 57% of the 2005 lubricant use, more than half being in commercial fleet and off-road vehicles (29%) and the rest in passenger cars and vans (28 %). The

balance of 43% is used in nonautomotive (industrial) applications. World lubricant use by product type is provided in Figure 1.2. It is important to note that the use ratio between the two types of applications varies across regions. For example, automotive use in Western Europe is 47% and in the relatively less industrialized nations of the Near/Middle East it is as high as 74%. The U.S. lubricant growth in recent years has been slow, on the order of around 1%. The slow growth of the automotive lubricants in the United States can be ascribed to smaller crankcases in newer cars, decrease in the distance driven per year, reduction in new U.S. made car purchases, and tighter modern engines which consume less oil per traveled mile. The sluggish growth in industrial lubricants reflects the general trend of the United States away from being a heavy industry based economy. Historical data show that the overall global lubricant market has not changed much over the last decade. This is because the increased demand in Asia and Latin America is offset by a drop in demand in Western Europe and North America. The additional factors that are either affecting the present lubricant demand or will impact future consumption of the lubricants, hence the additives, include the following:

- New engine designs to achieve more efficient combustion.
- Continuously variable transmissions (CVTs).
- On-board oil monitoring.
- New performance classifications or specifications.
- Government regulations pertaining to the stringent emissions standards and lowering of the certain chemical elements, such as phosphorus, chlorine, and some metals, in lubricants due to their real or perceived adverse effects on the environment.
- Extended service intervals.
- Filled-for-life, sealed-for-life drive lines.
- Fuel cell technology.
- Development and or growth of electric cars and hybrid cars (Rizvi, 2009).

The last decade has seen a slow but steady move toward the use of environmentally friendly or more readily biodegradable lubricant fluids. Biodegradability has become one of the most important design parameters both in

the selection of the base fluid and in the overall formulation of the finished lubricant. By more readily biodegradable it is meant that the fluids, using Standard methods and assays, are converted from the lubricating fluids to lower molecular weight components that have essentially no environmental impact. The rate at which lubricants, and other chemicals or additive components, biodegrade is related to their chemical structure. Their chemical structure affects their properties, many of which affect performance in the various tests for biodegradability. For example, water solubility is critical in some tests for biodegradability, and toxicity is very important, because if the lubricant is toxic and reduces the organism population, then this directly and negatively impacts the process of biodegradation (Rudnick and Erhan, 2006).

One of the primary choices of base oils for environmentally aware (EA) lubricants today is vegetable oils. This is due to their good natural biodegradability and very low toxicity in combination with very good lubricity characteristics. These renewable resources also provide a cost advantage over other EA base materials such as synthetic base stocks. But, there are some performance limitations of the vegetable-based lubricants that have been and continue to be addressed. These characteristics are mainly high temperature oxidation stability, low temperature performance, viscosity limitations, and cost. Although less expensive than synthetic alternatives, vegetable-based products can cost several times as much as conventional mineral-based lubricants. Genetic engineering will provide improved performance in the future in areas of oxidation stability and low temperature performance by increasing the high oleic acid content as well as other by means of genetic alterations (branching). In most applications, the vegetable-based EA lubricants can be formulated to perform in all but the most severe equipment. It is important to note that not all vegetable-based EA lubricants will provide the same levels of performance. Vegetable-based oils derived from rapeseed plants, cotton seeds, soybean oil, sunflower seed oil, corn oil, palm oil, and peanut oils are frequently used materials, with rapeseed being the most common (Pirro and Wessol, 2001).

In this thesis, the work is performed on Jojoba Oil (JO) and its lubricating oil performance characteristics. JO has good lubricity and it can be utilized as a component in lubricating oil formulations. The physicochemical properties of the oil when compared with those of mineral oil base stocks showed that the pour point, acid value and oxidative stability were the limiting factors in its use as a base stock. Studies were carried out to improve these properties. The results indicated that these properties can be modified by physical and chemical methods and also by additive treatment.

2. PREVIOUS STUDIES

2.1. Bio-Based Lubricants

Compared to the lubricants made of petroleum, vegetable-based lubricants are much more biodegradable but inferior in many other technical characteristics. The base stock typically contributes to more than 80% of lubricant and must meet performance criteria in such aspects as cleanliness, viscometric properties, volatility, oxidative and hydrolytic stability, deposit forming tendencies, solvency, miscibility or compatibility with system elastomers and other. For vegetable-based lubricants, oxidative stability and low temperature problems are considered the most critical. Thin film oxidation test was used to compare oxidative stabilities. Vegetable oils appear an order of magnitude less stable than mineral oils or synthetic biodegradable basestocks, such as isoalkyl adipates or polyalphaolefins. Low temperature performance of vegetable oils, namely pour points and cold storage, was also problematic. These problems can only partially be relieved by lubricant additives, thus vegetable oils have to be modified chemically to eliminate sites susceptible to oxidation and to disrupt formation of crystals at low temperatures (Erhan and Asadauskas, 2000).

The development of lubricants like, e.g. engine and hydraulic oils was traditionally based on mineral oil as a base fluid. This fact is related to the good technical properties and the reasonable price of mineral oils. The Report to the Club of Rome (W.W. Behrens III, D.H. Meadows, D.I. Meadows, J. Randers, The limits of growth, A Report to the Club of Rome, 1972) and the two oil crises of 1979 and 1983, however, elucidated that mineral oil is on principle a limited resource. In addition, environmental problems associated with the production and use of chemicals and the limited capacity of nature to tolerate pollution became obvious (G.H. Brundtland, et al., in: Hauff, Volker (Ed.), World Commission on Environment and Development (WCED), Report of the Brundtland-Commission, Oxford, UK, 1987), and the critical discussion included besides acid rain, smog, heavy metals, and pesticides also mineral oil (especially oil spills like the case Exxon Valdes). A

disadvantage of mineral oil is its poor biodegradability and thus it's potential for long-term pollution of the environment. From the early development of lubricants for special applications (e.g. turbojet engine oils) it was known, those fatty acid polyol esters have comparable or even better technical properties than mineral oil. Subsequently, innumerable synthetic esters have been synthesized by systematic variation of the fatty acid and the alcohol components. Whereas the alcohol moiety of the synthetic esters is usually of petrochemical origin, the fatty acids are almost exclusively based on renewable resources. The physico-chemical properties of oleochemical esters can cover the complete spectrum of technical requirements for the development of high-performance industrial oils and lubricants (e.g. excellent lubricating properties, good heat stability, high viscosity index, low volatility and superior shear stability). For a comprehensive review of their technical properties see F. Bongardt, in: Jahrbuchfür Praktiker, H. Ziolkowsky (Ed.), Verlag für Chemische Industrie GmbH, 1996, pp. 348-361. This article will focus on the ecological properties of oleochemical (synthetic) esters. The environmental relevance of oleochemicals in comparison to petrochemicals is discussed, and then the principles of an ecological assessment are described. The ecotoxicological properties and the biodegradability of oleochemical esters are presented. Finally, the ecological properties of the oleochemical esters are discussed with regard to existing environmental classification and labeling systems (Willing, 2001).

JO and other vegetable oils, such as soybean, sunflower and castor oils, were evaluated to be used as lubricants. Three standard mineral lubricating oils were considered in this study as reference. The essential parameters tested for comparisons were the oil viscosity, viscosity index, and viscosity-temperature and shear rate-shear stress relationships. The effect of excessive heating on the vegetable oils' stability was studied and the corresponding parameters were also measured. JO was found to be the best among all tested oils, whereas it gave the minimum change in viscosity gradient and hence the highest viscosity index. There was a linear relation between shear rate and shear stress of all oils before and after heat deterioration. Therefore, these oils were considered as Newtonian liquids. However, the oil viscosities as well as the rate of viscosity variation with temperature were affected by heat deterioration

being lowered in value, in case of JO, and higher value, in case of castor oil. JO was examined for other important properties for its use as a lubricant, such as refractive index, acid value, peroxide value, saponification value, iodine value, flash, fire and pour points (El Kinawy, 2004).

Boundary lubrication in a dynamic mechanical system is primarily governed by the formation of a stable tribochemical film. Polar functional groups in the triacylglycerol molecule of vegetable oil in conjunction with oil-additive-metal interaction during the metal rubbing process can significantly improve the wear resistance and extreme-pressure lubrication. Increasing the polar functionality of vegetable oil structure has a positive impact on wear protection resulting from stronger adsorption on metal surface as well as greater lateral interaction between the ester chains. The results reveal that ester groups of triacylglycerol molecule react with phosphate groups to form a protective fatty-phosphite layer. Antimony impregnation into the metal surface following strong complexation reaction with metal sulfide results in significant improvement in extreme-pressure characteristics due to alteration in surface metallurgy. In this case, seizure is averted even when the fluid adsorbed layer and phosphite coating is removed because of antimony induced surface hardness. The difference can be observed by analyzing the metal surface using scanning electron microscope (SEM). Selected bio fluids viz. soybean oil (SBO), thermally modified soybean oil (TMSBO) and chemically modified soybean oil (CMSBO) were investigated for potential application as industrial fluids (Adhvaryu et al., 2004). Vegetable oils are being investigated as a potential source of environmentally favourable lubricants, due to a combination of biodegradability, renewability and excellent lubrication performance. Low oxidation and thermal stability, poor low-temperature properties and narrow range of available viscosities, however, limit their potential application as industrial lubricants. This review addresses oxidation as a limitation of vegetable oil-based lubricants. The basic mechanism of vegetable oil autoxidation is presented, along with methods used to monitor and analyze the products of oxidation. The potential impact of such oxidation products on lubrication performance is discussed. A brief discussion of

methods used to assess and improve oxidation stability completes the review (Fox and Stachowiak, 2007).

Future lubricants have to be more environmentally adapted, have a higher level of performance, and lower total life cycle cost (LCC) than presently used lubricants. To be able to formulate those lubricants, the properties of the base fluids have to be well known. Base fluid properties that influence the formulated lubricant performance could be divided into three different groups. These groups are: physical, chemical, and film formation properties. In this study, properties from all of these groups are investigated to improve the understanding on their influence on base fluid overall performance.

There are more or less environmentally adapted base fluids available for formulation of lubricants. They could be divided into different groups, mineral, semi-synthetic, and synthetic fluids. Synthetic fluids could be of different types: polyalpha olefins (PAO), synthetic ester, polyglycols, and others. The most interesting group for formulation of environmentally adapted lubricants are the synthetic esters. In this study, the properties for a large number of environmentally adapted ester base fluids are studied in detail. The tested properties relate to the macroscopic/molecular behavior and include: viscosity-temperature-pressure effects, η (p, T), thermal conductivity, λ (p, T), and heat capacity per unit volume, ρ c_p (p, T). The film formation capability in elasto-hydrodynamic contacts is also studied. Different connections between the molecular structure and the performance of the fluids are discussed. As an example, it is found that a large number of carboxylate groups in the ester molecule improve the thermal properties, and thereby a thicker lubricating film could be maintained in highly loaded, high-slip contacts (Pettersson, 2007).

The study describes the tribochemical evaluation of vegetable oil based antiwear additive obtained through chemical modification. The Sulfur was incorporated using a chemical reaction of epoxidized vegetable oil and common thiols, resulting in formation of a hydroxy thioether derivative of vegetable oils. The synthesis retains the vegetable oil structure, eliminates poly-unsaturation in the molecule, and adds polar functional groups that significantly improve adsorption on metal surfaces. These additives are obtained by chemical modification of oils

originated from natural resources. The tribochemical behavior of sulfur incorporated vegetable oil was studied by measuring friction coefficient using ball-on-disk configuration and wear scar diameter using four-ball configuration. Comparative tests with commercial antiwear additives demonstrate the effectiveness of these derivatives. The derivatives were found useful as agriculturally based antiwear additives for lubricant applications (Sharma et al., 2009).

2.2. Jojoba Oil as a Lubricant

JO has good lubricity and it can be utilized as a component in lubricating oil formulations. The physicochemical properties of the oil when compared with those of mineral oil base stocks showed that the pour point, acid value and oxidative stability were the limiting factors in its use as a base stock. Studies were carried out to improve these properties. The results indicated that these properties can be modified by physical and chemical methods and also by additive treatment. It has also been possible to increase the viscosity of JO by partial sulphurization, resulting in widening the scope of its utilization in developing lubricant formulation (Bhatia et al., 1990).

In order to develop JO based extreme pressure (EP) additives, a number of sulphurized JO have been prepared by varying the reaction conditions. The products differ in their amount of sulphur and in their structure. An attempt to predict their performance based on their structure reveals that it is not a simple addition of sulphur which is required; a certain level of cross linking and polymerization also are desirable to have a product which is effective as an EP additive (Bhatia et al., 1998).

Viscosity, flash point, refractive index, viscosity index, specific gravity, aniline point, foam testing, color stability, ash content, water content, saponification value, iodine value, and other properties were investigated for Jordanian JO. Results showed that JO has low ash and water contents and high flash point and viscosity index. Viscosity and specific gravity changed only slightly with temperatures. As an additive, JO improved the viscosity index of lubricants from 100 to 130. When subjected to heating and cooling from 40 to 200°C, the chemical structure, kinematic

viscosity, and refractive index remained almost constant. Thus, JO was highly stable in this temperature range (Allawzi et al., 1998).

JO extraction by pressing alone, pressing followed by leaching, and leaching alone were investigated. The extraction process by first and second pressing followed by leaching gave about 50% by weight oil with reference to total seed, which is in agreement with what has been reported previously. The extraction by leaching process was carried out using different solvents. These solvents were; hexane, benzene, toluene, petroleum ether, chloroform, and isopropanol. Hexane, benzene, and petroleum ether gave the highest yield (all about 50% by weight oil with reference to total seed), but when cost is considered, petroleum ether is recommended as the best solvent to leach JO. The yield obtained in this work for leaching by hexane and benzene are 3-5% and about 10% for isopropanol more than those reported in the literature. Traces of solvent remained with the extracted oil after simple distillation followed by a second stage distillation via a Rotavapour apparatus. These traces slightly affected some of the oil properties such as pour point and flash point (Abu-Arabi et al., 2000).

The aim of the work was to evaluate the effect of extraction method and bleaching on the quality of jojoba waxes from Argentina. Jojoba waxes obtained by cold pressing, Golden wax (expeller-pressed wax) and Lite wax (bleached wax) were analyzed using standard methods adopted as recommended practices by the American Oil Chemists Society. Physical parameters, fatty acid and alcohol compositions were unchanged among waxes. Cold-pressed wax was noteworthy by its lower peroxide value, higher amounts of tocopherol and total phenol contents. Accordingly, it presented the best oxidative stability. Bleaching caused a strong decrease in both tocopherol and phenol contents; consequently the bleached wax showed poor oxidative stability (Tobares et al., 2003).

Liquid wax ester extracted from the seeds of the jojoba plant (Simmondsia chinensis) is used commercially in the cosmetics industry. For this reason, it is a potentially profitable crop for the arid regions of Chile. This study evaluates the yields, and physical and chemical properties of jojoba seeds grown experimentally in Chile, compared with literature values and commercial sources in the United States,

Israel, Peru, and Argentina. Cuttings from promising jojoba plants were gathered from abandoned plantations in various arid regions of Chile and propagated for study at a Chilean research station. Seed samples originating from 17 selected clones were analyzed for oil content, weight per 1000 seeds, and protein and simmondsin contents. The seed oil was analyzed for acid value, wax ester profile, fatty acid profile and fatty alcohol profile. The Chilean seeds had physical properties similar to commercial sources and literature values. However, there were differences in the chemical composition. The molecular weights of the wax esters and their fatty acid moieties were significantly higher than the literature values and commercial seed samples. The wax ester profile of the Chilean seeds had higher C_{42} and lower C_{38} content than the commercial sources. In addition, the total simmondsins content in the Chilean seed was higher than literature values and the simmondsin content with respect to its analogs (simmondsin ferulate, demethylsimmondsin (DMS), and didemethylsimmondsin (DDMS)) was considerably lower (Cappillino et al., 2003).

In the study, hexane is used as solvent to extract JO in a batch leaching process. The effects of temperature, mixing speed and particle size and solvent-to-seed ratio on JO extraction were investigated. The best recovery of oil was obtained when the parameters investigated were at the boiling point temperature of the solvent, a particle size of 0.48 mm, a mixing speed of 1000 rpm, and a solvent-to-seed ratio of 15 ml/g. The maximum amount extracted was 55% wt under the above-mentioned operating conditions. The amount extracted at a higher particle size (1.5 mm) was around 44% wt. An empirical model was obtained based on the experimental data, in order to predict the extraction yield under the operating conditions. The derived model is in good agreement with the experimental data, with a maximum deviation of 8% (Allawzi et al., 2005).

Improvement of the oxidative stability of soybean oil (SBO) by blending with JO was investigated. SBO in the presence of 5, 10, 15 and 20% wt of JO was subjected to accelerated storage at 60°C. Peroxide values (PV), anisidine values (AV), UV absorption characteristics (K_{232} and K_{270} values), and headspace volatiles were determined to monitor the oxidative stability of oil samples. JO was effective in reducing the formation of hydroperoxides and volatile compounds in SBO. The

effect was remarkable in SBO/JO blends containing 15 and 20 % JO, which showed significant reductions in PV, AV and volatile content with respect to pure SBO. The increased oxidative stability of SBO/JO blends could not be attributed to JO tocopherols, since the addition of JO to SBO significantly reduced the tocopherol content of SBO. Besides the tocopherol content and unsaturation degree of SBO and JO, the effect of the JO ester structure on the oxidative stability of the blends is discussed. The enhanced chemical and flavor stabilities of SBO/JO blends with respect to pure SBO may make a significant contribution to improve the shelf life of SBO by reducing the deterioration reactions related to lipid peroxidation (Torres et al., 2006).

3. MATERIAL AND METHOD

3.1. Material

3.1.1. Jojoba Seeds

Jojoba (Simmondsia chinensis) is a dwarf, evergreen perennial shrub, olive green in color. Besides its superior lubricating properties, jojoba has attracted interest as a landscape and soil conservation plant. The species is dioecious and the seedlings cannot be sexed until the first flower buds appear 9 to 24 months after sowing. Although jojoba plants start producing fruit in 3 years, full maturity takes 10 to 12 years, with the plants life estimated to be 100 years.

Its origin is the Sonora desert region of Arizona and California in the USA and the states of Sonora and Baja California in Mexico.

Jojoba plants 3 or more years old can usually survive winter temperatures as low as -10^{o}C; however many flower buds are killed by temperature -4 to -7^{o}C or lower.

The oil concentration of the seed at maturity ranges from 38 to 62% of seed dry weight and oil is obtained by pressing or by solvent extraction. JO is a natural mimic of the oil secreted by human skin and so it may be used to protect and lubricate the skin and hair. JO may also be used in disinfectants, detergents, emulsifiers and dryers, plasticizers protective coatings and corrosion inhibitors (Ulger et al., 2002).

In this study jojoba seeds were obtained from Çukurova University Department of Field Crops. These seeds were cultivated on the university farmland. They have grain size values between 9 and 13 mm (Figure 3.1.). Jojoba seeds were separated from stones and plant parts. This provides a more constant oil quality and reduces the risks of damaging the knives of the crushing mill.



Figure 3.1. Jojoba seeds

3.1.2. Jojoba Oil

The Jojoba Oil was extracted from the seeds of the jojoba, perennial shrub that grows in Çukurova region. Chemical properties of JO are shown in Table 3.1. Jojoba oil, is a mixture of straight-chain esters of monounsaturated, long-chain fatty acids and long-chain primary fatty alcohols, mainly two ester molecules containing 40 and 42 carbon atoms representing the 80% of the oil.

Table 3.1. Chemical Properties of Jojoba Oil

Retention Time	% Area	Compound
45,25	0,805811	Hexadecanoic Acid Methyl Ester
49,46	4,805332	Ethyl Oleate
49,95	0,518675	Octadecanoic Acid Methyl Ester
54,09	14,71952	Oleyl Alcohol
55,35	52,44268	11-Eicosenoic Acid Methyl Ester
62,31	14,79321	Eicosen-1-Ol,Cis-9-
63,37	9,386592	Methyl Erucate
68,69	1,594871	(2)-14-Tricosenyl Formate
69,51	0,933308	Oleic Acid

3.1.3. Mineral Base Oils

A modern lubricant comprises a base fluid and chemical additives. A vast majority of lubricants are based on mineral oils, which is because of their low cost, ready availability, and overall adequate performance. Since the base fluid makes up the bulk of the lubricant, its properties greatly impact the properties of the lubricant. The properties of a mineral base stock depend on its source, viscosity, and the degree of refining. As stated before, mineral base stocks or oils are classified on the basis of the predominant hydrocarbon type present in their composition, that is, as paraffinic, naphthenic and aromatic, or bright stocks. Unfortunately, the hydrocarbon-based classification is not clear cut but is vague because these base stocks are manufactured based on viscosity and not on structural composition. Hence, neither base stock is purely paraffinic, naphthenic, or aromatic, but is a blend of all three types of hydrocarbons. While each type of hydrocarbons has its own advantages and disadvantages, to formulate lubricants for most applications, the paraffinic base stocks are often preferred. However, they do suffer from poor low-temperature performance because of the wax formation, which incidentally is corrected by the use of additives, called pour point depressants. Naphthenic base stocks are the next best in desirable overall properties. While these base stocks have mediocre viscosity indices and oxidative stability, they have good viscosity pressure relationship and good low-temperature properties. Aromatics are the least suited to formulate most modern lubricants, but they do have good solvency, thermal stability, and viscosity-pressure properties. Their oxidation stability and VI characteristics are the poorest among the three groups. As a general rule, the base oils of different origin may be similar in many respects, but they subtly differ in some properties, such as oxidation stability, which are determined by their overall structural composition. Desirable base oil properties include good oxidation resistance, high viscosity index, good low-temperature performance, and low volatility. The refining process has the flexibility to produce base stocks where these properties are optimized. However, the challenge is to achieve this cost effectively. Good quality crudes, such as those from the Middle East, help accomplish this manufacturing goal. Base stock production results

in a number of products based upon viscosity. These base stocks are blended to make base oils of the desired viscometrics. The resulting base oils do not always possess the optimal properties for use in a specific application. Hence, they need performance-enhancing additives. Some additives enhance physical properties of the base oils while others impart or improve their chemical properties and behavior.

Untreated or nonformulated lubricants (mineral base oils and synthetic base stocks) do not possess the necessary properties to perform effectively in today's demanding lubricating environments. To function properly in such environments, base fluids need the help of chemicals, called additives. Additives improve the lubricating ability of the base oils either by enhancing the desirable properties already present or by adding new properties. Most of today's lubricants are formulated lubricants, and additives are their integral part. The world consumption of lubricant additives has increased from 2.6 million metric tons in 1997 to about 3 million metric tons in 2006. The consumption is expected either to plateau or grow slowly in North America and Western Europe, which consume the largest share of the total. The developing economies of Asia, such as India and China, and of Latin America, such as Brazil, Chile, and Argentina, are expected to see a faster growth. A formulated lubricant comprises a base fluid and a performance package, and in the case of multi-grade oils, an additional viscosity modifier. The amount of the base fluid in a lubricant can be anywhere from 70% to greater than 99%, based on the desired performance level and the severity of the end-use requirements. Base fluid is derived from three sources: petroleum, synthetic, and biological, i.e., plant or animal in origin (Rizvi, 2009).

Four types of mineral base oils, named spindle oil (SO), light neutral (LN), heavy neutral (HN), bright stock (BS) were obtained from Gema Mineral Oil Co. Inc. The viscosity values of the selected oils are tabulated in Table 3.2. and the characteristics of these mineral base oils are explained below.

Table 3.2. Viscosity values of oils used in experiments

Type of Oil	**Tüpraş Standards (cSt)**	**Viscosity Values at 100°C (cSt)**
Spindle oil [a]	3,2-4	3,95
Light neutral [b]	5-5,7	5,66
Heavy neutral [c]	10,4-11,6	11,05
Bright stock [d]	32-36	34,26

[a] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=900&lRedirectPageID= 145
[b] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=910&lRedirectPageID=145
[c] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=930&lRedirectPageID=145
[d] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=940&lRedirectPageID=145

3.1.4. Crushing Mill

Since jojoba seeds have strong cover, before extracting the oil from them it is necessary to crack the seeds. This process also increases solvent contact area for the best extraction quality. During the experiments, jojoba seeds were cracked with crushing mill (Figure 3.2.) for two times to get more homogeneous seed thickness. The specifications of the crushing mill can be seen in Table 3.3.



Figure 3.2. Crushing mill

Table 3.3. Specification of crushing mill

Specifications	Values
Diameter of crashing chamber	160 mm
Feeding entrance	20x40 mm
Power of milling engine	1,1 kW
Speed of milling engine	3000 rpm
Working voltage	220 V

3.1.5. Soxhlet Extractor

In order to extract oil from jojoba seeds a soxhlet extractor was used. In a soxhlet extractor, solid material containing some of the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the soxhlet extractor. The soxhlet extractor is placed onto a flask containing the extraction solvent. The soxhlet is then equipped with a condenser. The solvent is heated to reflux. The solvent vapour travels up a distillation arm and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapor cools, and drips back down into the chamber housing the solid material. The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days. During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled. After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded (Anonymous, 2010).

Soxhlet extractor consists of 11 parts which are presented in Figure 3.3 and Figure 3.4.

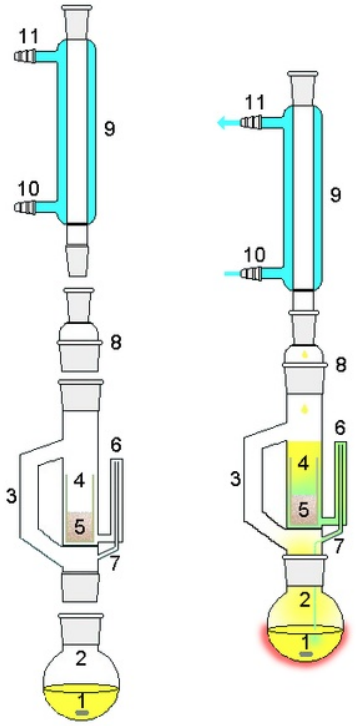


Figure 3.3 Schematic representation of soxhlet extractor

1. Stirrer bar/anti-bumping granules

2. Still pot (extraction pot) - still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the soxhlet chamber.

3. Distillation path

4. Soxhlet Thimble

5. Extraction solid (residue solid)

6. Syphon arm inlet

7. Syphon arm outlet

8. Expansion adapter

9. Condenser

10. Cooling water in

11. Cooling water out

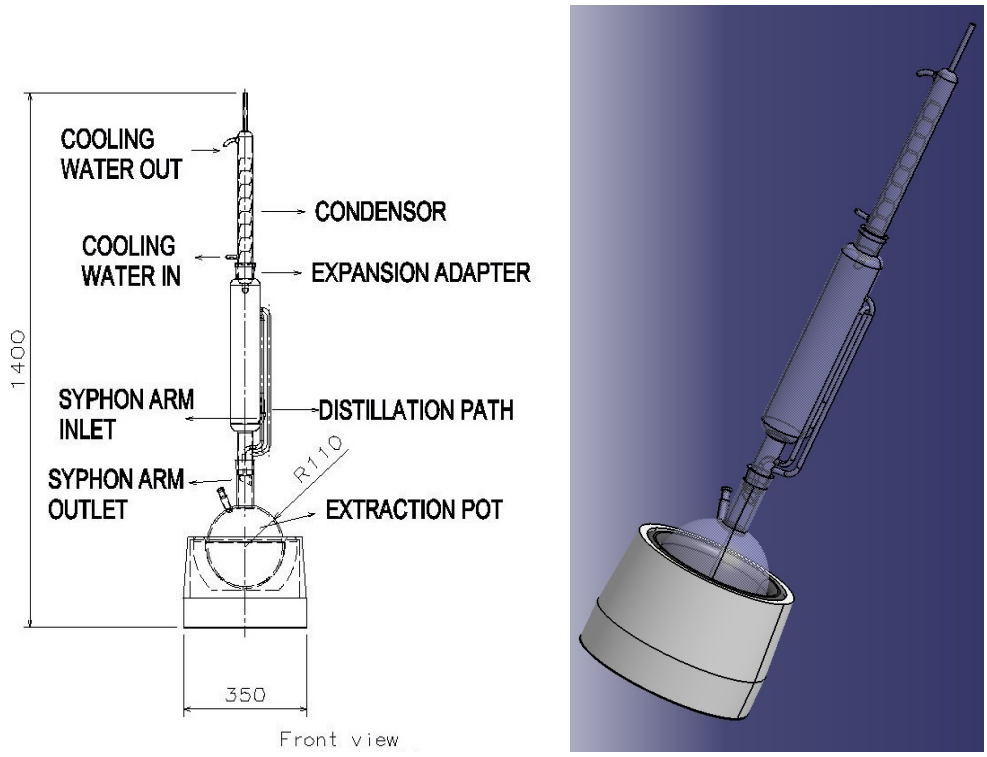


Figure 3.4. 3 dimensional view of soxhlet extractor

3.1.6. Hexane as a Solvent

n-Hexane is mainly used as a solvent in the oil extraction process for vegetable oil. n-Hexane (CAS No: 110-54-3) specifications are given in the Table 3.4.

Table 3.4. Specifications of n-Hexane

Specifications	**Approximate Values**
Purity	95 %
Density ($20^oC/4^oC$)	0.659-0.662 g/cm^3
Water	0.02 %
Boiling Point	69 ^oC

3.1.7. Hydrometer

A hydrometer is an instrument used to measure the specific gravity (or relative density) of liquids; that is, the ratio of the density of the liquid to the density of water and shown in Figure 3.5.

A hydrometer is usually made of glass and consists of a cylindrical stem and a bulb weighted with mercury or lead shot to make it float upright. The liquid to be tested is poured into a tall jar, and the hydrometer is gently lowered into the liquid until it floats freely. The point at which the surface of the liquid touches the stem of the hydrometer is noted. Hydrometers usually contain a paper scale inside the stem, so that the specific gravity can be read directly. The scales may be Plato, Oechsle, or Brix, depending on the purpose.

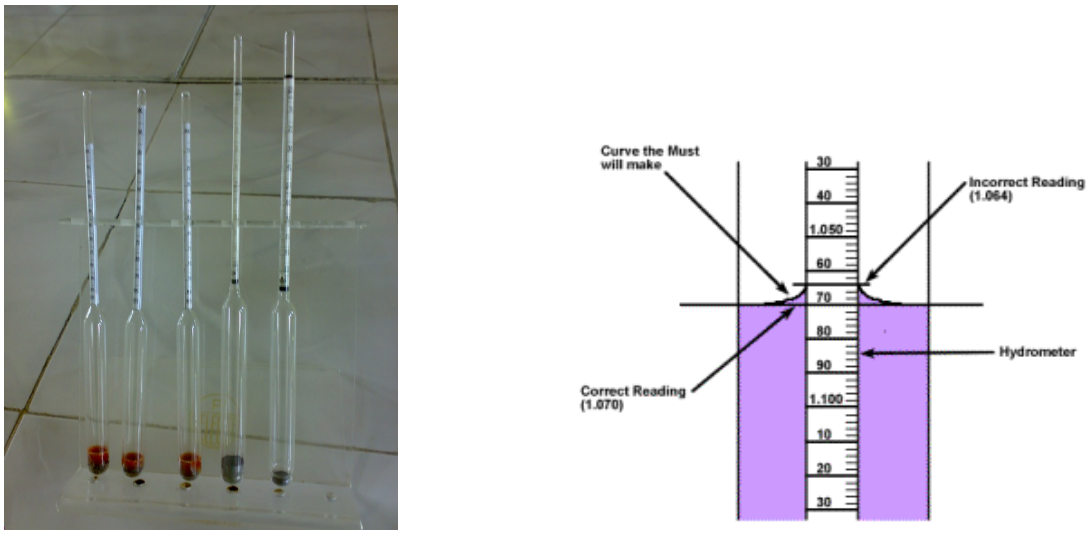


Figure 3.5. Different hydrometers and measuring method

3.1.8. Kinematic Viscosimeter

Model AKV-202 has been designed for automatic determination of kinematic viscosity. Once a specimen is set the series of processes including timing, cleaning/drying of viscosimeter tube, and reporting are fully automated. AKV-202 is

an economical bench-top system, which saves not only the technician's time but also valuable laboratory floor space and budget.

- Wide range of Kinematic Viscosity (KV) can be covered with the modified Lanz-Zeitfuchs viscosimeters. The reverse flow viscosimeter covers both transparent and opaque samples and also eliminates drainage error.
- Timing, sample draining, viscosimeter cleaning/drying, calculating/reporting are automated, saving the time of the experienced hands and improving repeatability.
- The two-viscosimeters/one-bath system is housed in a compact housing that is small enough to install on a lab bench.
- Viscometer is mounted to a viscosimeter holder and the viscosimeter/holder assembly can be removed from the bath at ease, which assures easy viscosimeter replacement.
- The viscosimeter has a provision of overflow, which eliminates errors from inconsistency in sampling volume as well as thermal expansion of the sample.
- Two tanks are provided for cleaning and drying the viscosimeters, which allows quick and efficient cleaning/drying by selecting the best chemicals for the sample.



Figure 3.6. Kinematic viscosimeter

3.1.9. Pour Point Analyzer

MPC-102 series has been designed for automatic determination of Pour Point (PP) and Cloud Point (CP) with small specimen size and shorter test cycle time while securing better test precision than the conventional manual methods. PP measurement is made utilizing a new ASTM D6749 on Standard Test Method for Pour Point of Petroleum Products test method namely Air Pressure Method, which yields eventually no bias against the conventional test method, repeatability/reproducibility of 1/2^{o}C and 2-3 times faster determinations. The epoch-making high accuracy justifies PP determination at 1^{o}C intervals, which can help increasing the yields in the process. The CP/PP mode executes a CP determination and then PP determination consecutively, which further improves the test throughput in the lab. Multiple-tests versions with 6 test heads and 3 test heads are also available for higher volume tests.

The typical repeatability and reproducibility are 1^{o}C and 2^{o}C respectively, when PP is determined at 1^{o}C intervals. This high precision attributes to the patented Air Pressure method, in which the disturbance to the formation of wax crystal structure through the test process is kept at a minimal and consistent level. With this high precision, PP can be determined at 1^{o}C intervals for more precise process control, and therefore a considerable savings in the process can be realized.

Just set up a sample, select a test mode and then press the START key. The sample is cooled at the steepest possible rate without affecting the formation/growth of wax crystal, which has been known to be a critical factor for PP/CP determination. The test cycle time is typically 1/3 to 1/2 of that of the conventional tilting methods.

Since the required sample volume is a mere 4,5 ml and the sample cup is a test-tube type removable jar, the sample handling is extremely easy. Use of Peltier Cells for sample cooling/heating made this mini tester not only compact in design but energy efficient. Depending on the temperature range, air, tap water or small chiller with anti-freeze suffices the cooling requirement. No methanol is required.



Figure 3.7. Pour point analyzer

3.1.10. Timken Test Device

The tester (Figure 3.8.) is operated with a steel test cup rotating against a steel test block. The rotating speed is 123.71 ± 0.77 m/min which is equivalent to a spindle speed of 800 ± 5 rpm.

Two determinations are made: the minimum load (score value) that will rupture the lubricant film being tested between the rotating cup and the stationary block and cause scoring or seizure; and the maximum load (OK value) at which the rotating cup will not rupture the lubricant film and cause scoring or seizure between the rotating cup and the stationary block.

The values stated in SI units are to be regarded as standard. Because the equipment used in this test method is available only in inch-pound units, SI units are omitted when referring to the equipment and the test specimens.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

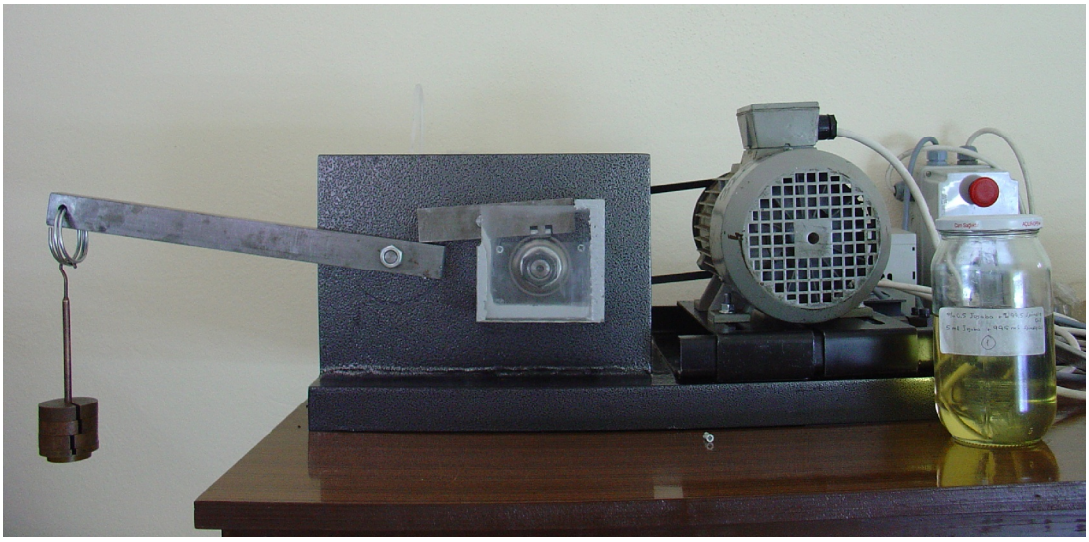


Figure 3.8. Timken test device

Extreme pressure (EP) additive in a lubricant is a substance that minimizes damage to metal surfaces in contact under high-stress rubbing conditions.

Scoring is a severe form of wear characterized by the formation of extensive grooves and scratches in the direction of sliding. Wear is damage to a solid surface generally involving progressive loss of material, due to relative motion between that surface and a contacting substance or substances. Load-carrying capacity of a lubricant is determined by this test method, the maximum load or pressure that can be sustained by the lubricant (when used in the given system under specific conditions) without failure of the sliding contact surfaces as evidenced by scoring or seizure or asperity welding. OK value is determined by this test method, the maximum mass (weight) added to the load lever weight pan, at which no scoring or seizure occurs. Score value is determined by this test method, the minimum mass (weight) added to the load lever weight pan, at which scoring or seizure occurs.

When the lubricant film is substantially maintained, a smooth scar is obtained on the test block, but when there is a breakdown of the lubricant film, scoring or surface failure of the test block takes place, as shown in Figure 3.9. and Figure 3.10.

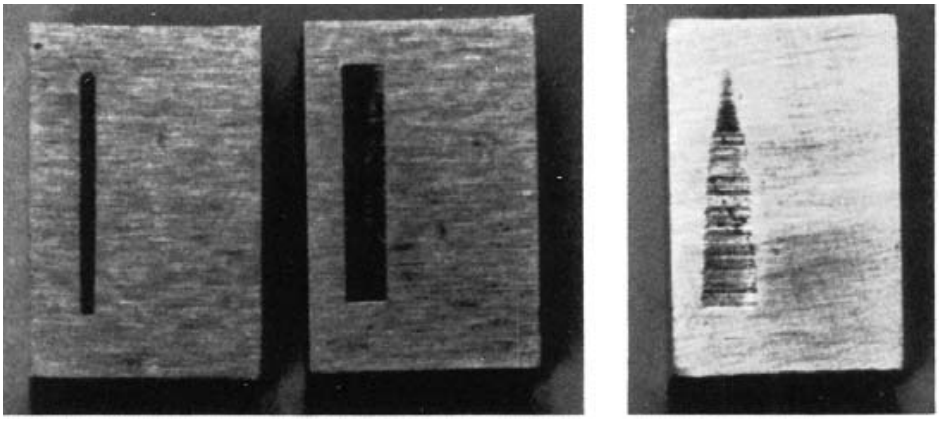


Figure 3.9. Test Blocks Showing Various Types of Scar

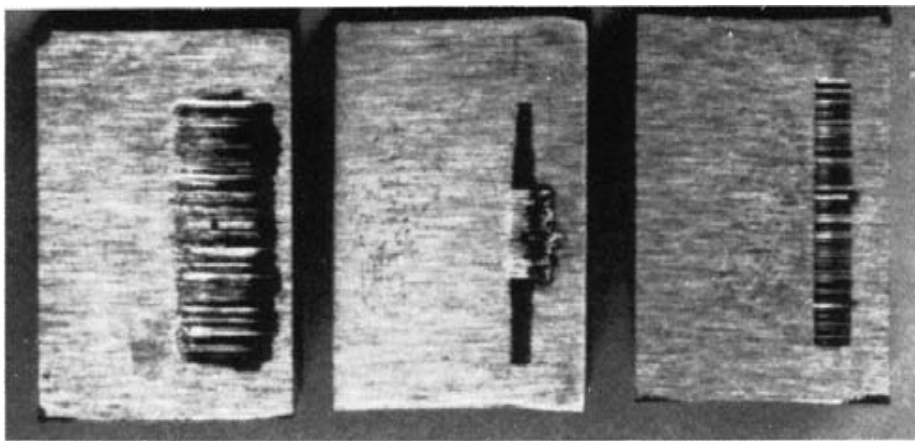


Figure 3.10. Scoring

Scoring is characterized by the furrowed appearance of a wide scar on the test block and by excessive pick-up of metal on the surface of the test cup. The form of surface failure more usually encountered, however, consists of a comparatively smooth scar, which shows local damage that usually extends beyond the width of the scar. Scratches or striations that occur in an otherwise smooth scar and that do not extend beyond the width of the scar are not considered as evidence of scoring.

3.1.11. Timken Test Blocks

Cylindrical test blocks were used in this experiment. Blocks have 12 mm diameter and 18 mm height and 60 R_C hardness values.



Figure 3.11. Timken test blocks

3.2. Method

3.2.1. Jojoba Oil Extraction

The solvent extraction process was applied to jojoba seeds which were obtained after the cracking process to get JO. Soxhlet extractor is used to extract the oil from cracked jojoba seeds. Hexane is used as solvent to extract JO in soxhlet extraction system. Hexane was found to be the best solvent for extraction because of its high oil yield, with no tendency to deposit residue on standing. Also hexane is commonly used in the leaching process because of its relatively low cost and low toxicity (Bhatia et al. 1990, Abu-Arabi et al. 2000). Extraction was done at the boiling point of the hexane at 69^{o}C.

Separation of the oil from the solvent was conducted under atmospheric pressure. The soxhlet extractor used was operated at a solvent to cracked seed ratio of 5:1 and with 10-12 passes of solvent.

3.2.2. Bleaching of Jojoba Oil

In this procedure, jojoba wax obtained from soxhlet extraction process, as previously described put in the bleaching vessel as a 1000 ml glass beacker. The beacker was putted on a thermostated magnetic stirrer heater (Figure 3.10.). The bleaching clay and silica gel were added in 800 ml crude jojoba seed oil. The mixture was then heated with stirring for 3 h at the desired temperature. The hot oil, clay and silica gel mixture was filtered by filter paper.



Figure 3.12. Bleaching Jojoba Oil

3.2.3. Preparing the Oil Blends

In this study, 20 different oil blends were prepared by mixing JO to four types of mineral base oils at volumetric rates of between 1 to 10% (Table 3.4. and Figure 3.11.). In order to get a good homogenized mixture, oils were heated to 40^{o}C before preparing the oil blends.

Table 3.5. Jojoba contents of prepared oil blends

Sample Number	Samples	Jojoba (ml)	Base Oil (ml)	Total (ml)
1	%1JO+SO	10	990	1000
2	2,5%JO+SO	25	975	1000
3	%5JO+SO	50	950	1000
4	%10JO+SO	100	900	1000
5	%1JO+LN	10	990	1000
6	2,5%JO+LN	25	975	1000
7	%5JO+LN	50	950	1000
8	%10JO+LN	100	900	1000
9	%1JO+HN	10	990	1000
10	2,5%JO+HN	25	975	1000
11	%5JO+HN	50	950	1000
12	%10JO+HN	100	900	1000
13	%1JO+BS	10	990	1000
14	2,5%JO+BS	25	975	1000
15	%5JO+BS	50	950	1000
16	%10JO+BS	100	900	1000



Figure 3.13. Oil blends prepared for tests

3.2.4. ASTM Test Methods

3.2.4.1. Density Measurement

Density is defined as the mass (m) per unit volume (v) of a substance at a given temperature (t), (For liquids measurements are made at about temperature of 20 ^{o}C) it can be calculated from the fallowing equation.

$$p_t = m/v \ (kg.m^{-3}) \qquad (3.1)$$

Density, relative density (specific gravity), or API gravity is a factor governing the quality and pricing of crude petroleum. However, this property of petroleum is an uncertain indication of its quality unless correlated with other properties. Density is an important quality indicator for automotive, aviation and marine fuels, where it affects storage, handling and combustion.

Accurate determination of the density, relative density (specific gravity), or API gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes or masses, or both, at the standard reference temperatures during custody transfer.

This test method is most suitable for determining the density, relative density (specific gravity), or API gravity of low viscosity transparent liquids. This test method can also be used for viscous liquids by allowing sufficient time for the hydrometer to reach equilibrium, and for opaque liquids by employing a suitable meniscus correction.

When used in connection with bulk oil measurements, volume correction errors are minimized by observing the hydrometer reading at a temperature close to that of the bulk oil temperature (ASTM D1298).

3.2.4.2. Viscosity Measurement

The parameter which plays a fundamental role in lubrication is oil viscosity. Different oils exhibit different viscosities. In addition, oil viscosity changes with temperature and pressure and the thickness of the generated oil film is usually proportional to it. So, at first glance it appears that the more viscous oils would give better performance, since the generated films would be thicker and a better separation of the two surfaces in contact would be achieved. This unfortunately is not always the case since more viscous oils require more power to be sheared. Consequently the power losses are higher and more heat is generated resulting in a substantial increase in the temperature of the contacting surfaces which may lead to the failure of the component. For engineering applications the oil viscosity is usually chosen to give optimum performance at the required temperature. Knowing the temperature at which the oil is expected to operate is critical as oil viscosity is extremely temperature dependent. The viscosity of different oils varies at different rates with temperature. It can also be affected by the velocities of the operating surfaces (shear rates). The knowledge of the viscosity characteristics of a lubricant is therefore very important in the design and in the prediction of the behavior of a lubricated mechanical system.

3.2.4.2.1. Dynamic Viscosity

Consider two flat surfaces separated by a layer of fluid of thickness **h** as shown in Figure 3.12. The force required to move the upper surface is proportional to the wetted area **A** and the velocity **u**, i.e.:

$$F \alpha A \times u \tag{3.2}$$

Assume that the fluid film separating the surfaces is made up of a number of infinitely thin layers. Compare now two fluid films of different thickness made up of equispaced layers. If the surface velocity remains unchanged in these two cases then a single layer in the thicker film will undergo less relative sliding than in the thinner film. The velocity gradients for these two layers will be different. Since the thicker film contains more single layers, less force will be needed to shear a single layer so the viscous resistance will vary as the reciprocal of the film thickness **1/h**. The force needed to move the upper surface is thus proportional to:

$$F \alpha A \times u/h \tag{3.3}$$

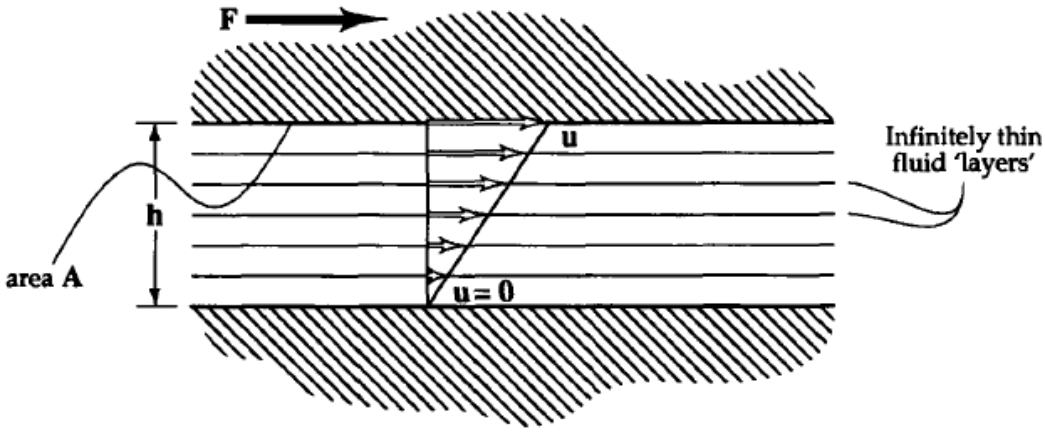


Figure 3.14. Schematic representation of the fluid separating two surfaces

This relationship is maintained for most fluids. Different fluids will exhibit a different proportionality constant **η** called the dynamic viscosity**.** The relationship (3.3) can be written as:

$$F = \eta \times A \times u/h \tag{3.4}$$

Rearranging gives:

$$\eta = (F/A) / (u/h) \tag{3.5}$$

or

$$\eta = \tau / (u/h) \tag{3.6}$$

η is the dynamic viscosity (Pas)

τ is the shear stress acting on the fluid (Pa)

u/h is the shear rate, i.e. velocity gradient normal to the shear stress s^{-1}

Before the introduction of the SI system the most commonly used dynamic viscosity unit was the Poise. Incidentally this name originated not from an engineer but from a French medical doctor Poiseuille, who studied the flow of blood. For practical applications the Poise (P) was far too large thus a smaller unit, the centipoise (cP), was more commonly used. The SI unit for dynamic viscosity is Pascal-second (Pas). The relationship between Poise and Pascal-second is as follows:

$$1 \text{ P} = 100 \text{ cP} \sim 0.1 \text{ Pas} \tag{3.7}$$

3.2.4.2.1. Kinematic Viscosity

Kinematic viscosity is defined as the ratio of dynamic viscosity to fluid density:

$$\upsilon = \eta / \rho \tag{3.8}$$

υ: is the kinematic viscosity (m^2/s)

η: is the dynamic viscosity (Pas)

ρ: is the fluid density (kg/m^3)

The most commonly used kinematic viscosity unit is the Stoke (S). This unit, however, is often too large for practical applications, thus a smaller unit, the centistoke (cS), is used. The SI unit for kinematic viscosity is (m^2/s), i.e.:

$$1\ S = 100\ cS = 0.0001\ m^2/s$$

The densities of lubricating oils are usually in the range between 700-1200 kg/m^3. The typical density of mineral oil is 850 kg/m^3. To find the dynamic viscosity of any oil in cP the viscosity of this oil in cS is multiplied by its density in g/cm^3, hence for a typical mineral oil:

$$\text{Viscosity in cP} = \text{viscosity in cS} \times 0{,}85\ g/cm^3$$

This test method specifies a procedure for the determination of the kinematic viscosity, ν, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer (ASTM D 445).

3.2.4.3. Pour Point Measurement

The pour point of oil (ASTM D97) is the lowest temperature at which the oil will just flow when it is cooled. In order to determine the pour point the oil is first heated to ensure solution of all ingredients and elimination of any influence of past thermal treatment. It is then cooled at a specific rate and, at decrements of 3^oC the container is tilted to check for any movement. The temperature 3^oC above the point at which the oil stops moving is recorded as the pour point. This oil property is important in the lubrication of any system exposed to low temperature, such as automotive engines, construction machines, military and space applications. When oil ceases to flow this indicates that sufficient wax crystallization has occurred or that the oil has reached a highly viscous state. At this stage waxes or high molecular weight paraffins precipitate from the oil. The waxes form the interlocking crystals which prevent the remaining oil from flowing. This is a critical point since the successful operation of a machine depends on the continuous supply of oil to the

moving parts. The viscosity of the oil at the pour point is usually very large, i.e. several hundred (Pas), but the exact value is of little practical significance since what is important is the minimum temperature at which the oil can be used (Stachowiak and Batchelor, 1993).

The pour point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, like pour point, can be critical for the correct operation of lubricating systems, fuel systems, and pipeline operations. Petroleum blending operations require precise measurement of the pour point. Test results from this test method can be determined at either 1 or 3^{o}C intervals. The standard procedure for measuring the pour point of oil is ASTM D 6749.

3.2.4.4. Timken Test Method

This test method covers the determination of the load carrying capacity of lubricating fluids by means of the Timken Extreme Pressure Tester.

This test method is suitable for testing fluids having a viscosity of less than about 5000 cSt (5000 mm^2/s) at 40^{o}C. The values stated in SI units are to be regarded as standard. SI units are omitted when referring to the equipment and the test specimens. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. The standard procedure for measuring the extreme pressure of oil is ASTM D 2782.

4. RESULTS AND DISCUSSION

4.1. Lubricant Properties of Oil Blends and Cost of Oils

This study was carried out at the Çukurova University Mechanical Engineering Department of Automotive Engineering Laboratories. Density, viscosity and pour point were the characteristics that tested according the standards. Timken life tests were performed. Lubricant quality testing results and standards were tabulated below (Table 4.1, 4,2).

Table 4.1. Lubricant quality standard test results

Type of Oil	Pour Point (^oC)(max.)	Viscosity Index (min.)	Viscosity (100^oC) cSt
Spindle Oil[a]	-12	94	3,2-4
Light Neutral[b]	-12	99	5-5,7
Heavy Neutral[c]	-9,4	94	10,4-11,6
Bright Stock[d]	-9,4	90	32-36

[a] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=900&lRedirectPageID= 145
[b] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=910&lRedirectPageID=145
[c] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=930&lRedirectPageID=145
[d] http://www.tupras.com.tr/detailpage.tr.php?strProductKey=940&lRedirectPageID=145

Table 4.2. Lubricant quality experimental test results

Type of Oil	Density (g/cm^3)	Pour Point (^oC)	Viscosity Index	Viscosity (100^oC) cSt
Spindle Oil[a]	0,856	-12	98	3,95
Light Neutral[b]	0,861	-11	101	5,66
Heavy Neutral[c]	0,872	-11	99	11,05
Bright Stock[d]	0,904	-13	90	34,26
Jojoba Oil	0,865	8	236	6,31

Table 4.3. Experimental and generally used motor oils' cost

Oils	Oil cost (TL/lt)	Explanation
Spindle Oil	2.3	Mineral Base Oil
Light Neutral	2.3	Mineral Base Oil
Heavy Neutral	2.3	Mineral Base Oil
Bright Stock	2.7	Mineral Base Oil
Jojoba Oil	15	Vegetable Oil (max. using rate 10%)
0W/30	28	Synthetic Motor Oil
5W/30	20	Synthetic Motor Oil
10W/30	16	Synthetic Motor Oil

Cost of mineral base oil, Jojoba oil which was used in experimental studies and 3 different synthetic motor oils that widely used in Adana are shown in Table 4.3. Compared to mineral base oils, Jojoba oil is more expensive but it is cheaper than synthetic motor oils. Addition of 10% of Jojoba oil to mineral base oil is in the range of economical limits.

4.1.1. Density

Lubricant density is an important specification in engineering calculations and sometimes offers a simple way of identifying lubricants. Density or specific gravity is often used to characterize crude oils. The density of JO is lower than that of Heavy Neutral and Bright Stock, which are 0,872 g/cm^3 and 0,904 g/cm^3 respectively. Due to the lower density of JO in accordance with the Heavy Neutral and Bright Stock, blending with JO was caused a reduction in the density of the blends (Figure 4.1, 4.2). Any increase in the percentage of the JO caused a decrease in the blend density. Also due to the higher density of JO in accordance with the Spindle Oil and Light Neutral, blending with JO increased the density of the blends (Figure 4.3, 4.4). Any increase in the percentage of the JO caused an increase in the blend density.

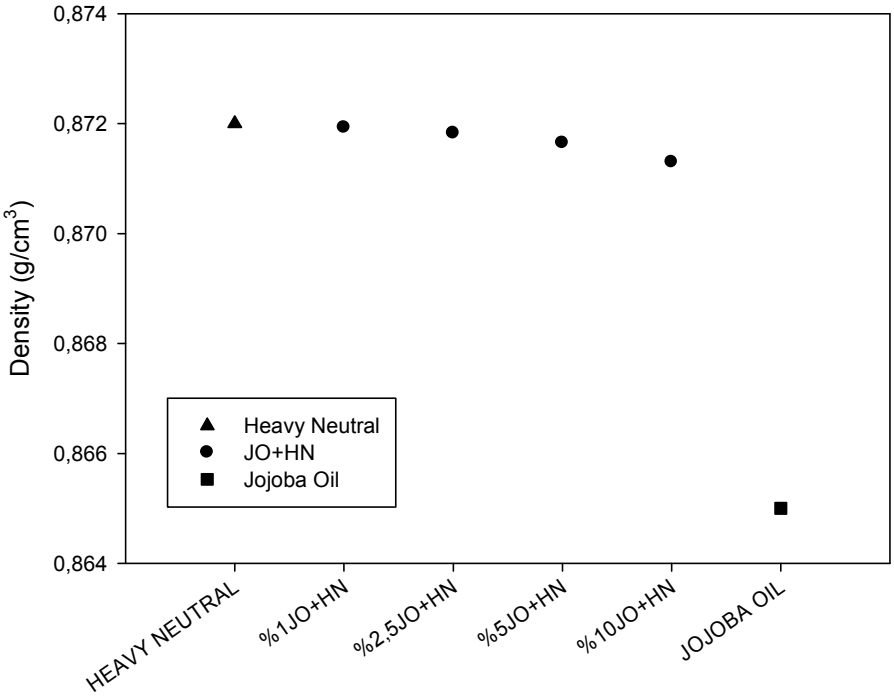


Figure 4.1. Effect of JO addition to density values of heavy neutral

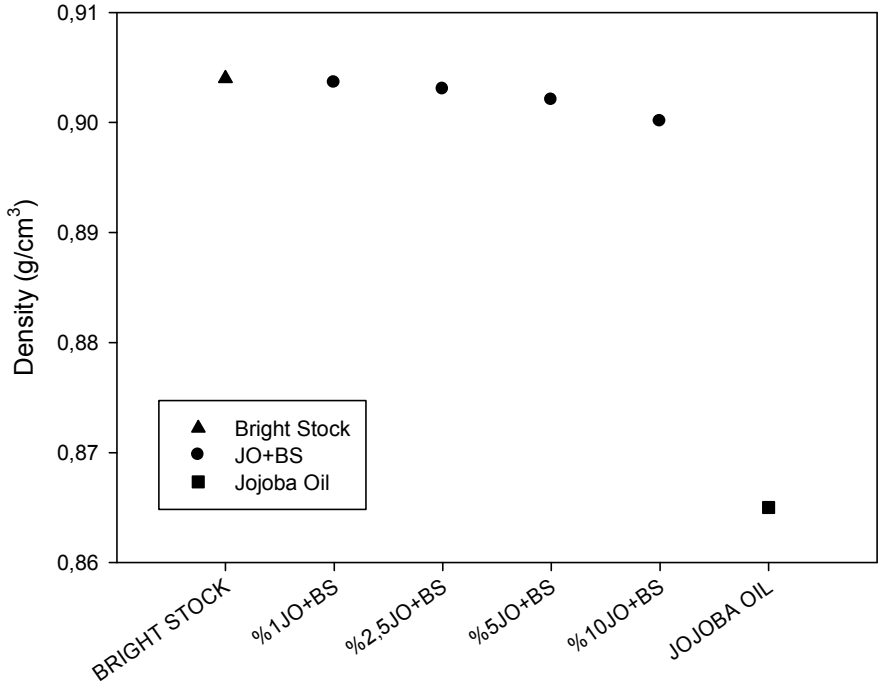


Figure 4.2. Effect of JO addition to density values of bright stock

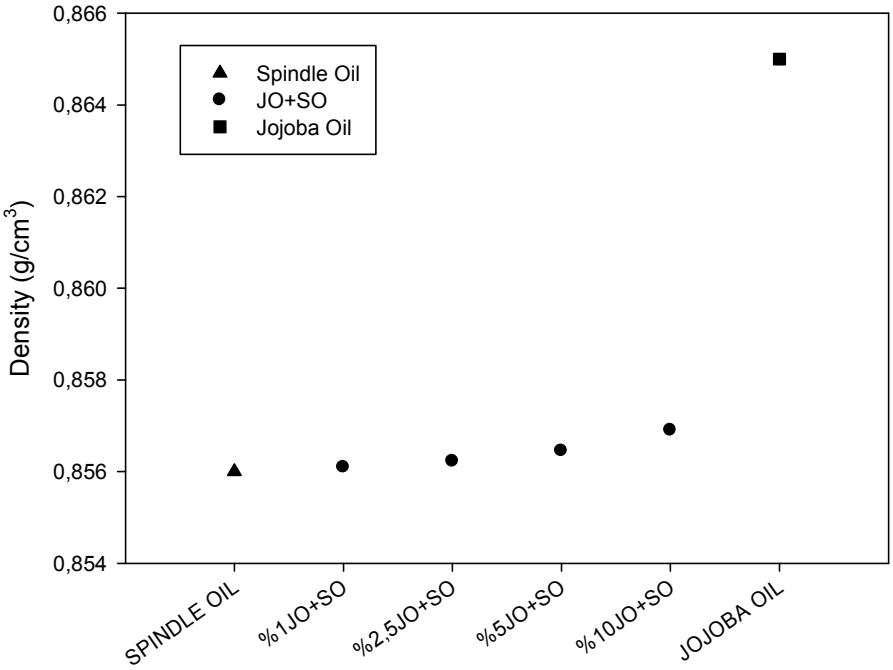


Figure 4.3. Effect of JO addition to density values of spindle oil

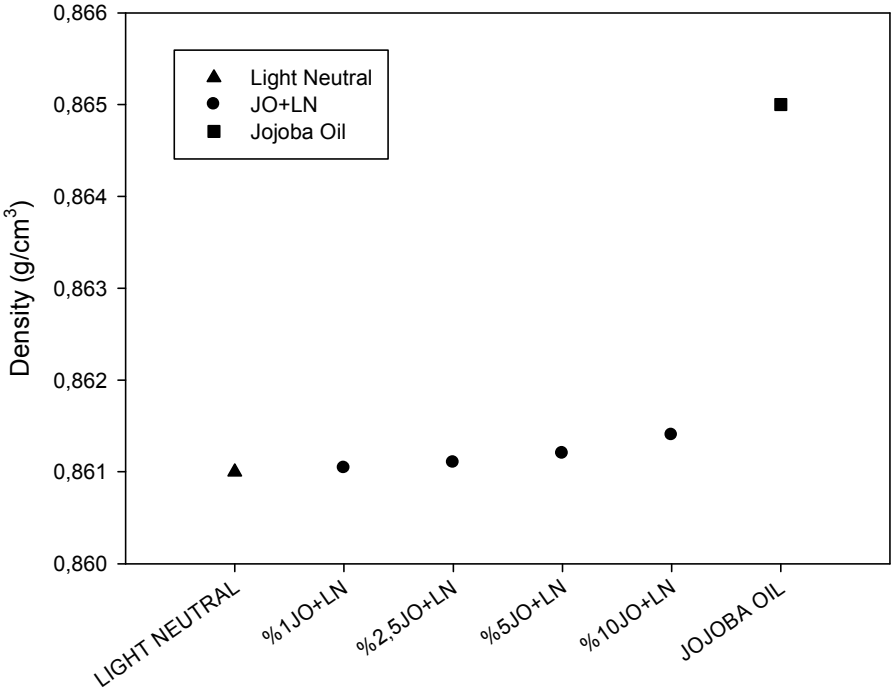


Figure 4.4. Effect of JO addition to density values of light neutral

4.1.2. Kinematic Viscosity

Probably the most important single property of lubricating oil is its viscosity. A factor in the formation of lubricating films under both thick and thin film conditions, viscosity affects heat generation in bearings, cylinders, and gears; it governs the sealing effect of the oil and the rate of consumption or loss; and it determines the ease with which machines may be started under cold conditions. For any piece of equipment, the first essential for satisfactory results is to use an oil of proper viscosity to meet the operating conditions.

In selecting the proper oil for a given application, viscosity is a primary consideration. It must be high enough to provide proper lubricating films but not so high that friction losses in the oil will be excessive. Since viscosity varies with temperature, it is necessary to consider the actual operating temperature of the oil in the machine. Other considerations, such as whether a machine must be started at low ambient temperatures, must also be taken into account.

The viscosity of any fluid changes with temperature increasing as the temperature is decreased, and decreasing as the temperature is increased. Thus, it is necessary to have some method of determining the viscosities of lubricating oils at temperatures other than those at which they are measured. This is usually accomplished by measuring the viscosity at two temperatures, then plotting these points on special viscosity temperature charts developed by ASTM. The two temperatures most used for reporting viscosities are $40^{o}C$ and $100^{o}C$ (Pirro and Wessol, 2001).

It was observed that the variation in the kinematic viscosity of the oil blends were depends on the density values of the oil blends. JO addition to Spindle Oil and Light Neutral were caused minor increments in the oil blends (Figure 4.5, 4.6) beside this the addition of the oil to Heavy Neutral and Bright Stock were caused minor decrement in the oil blends (Figure 4.7, 4.8). According to the higher kinematic viscosity value of the JO than the Spindle Oil and Light Neutral, kinematic viscosity of oil blends was increased.

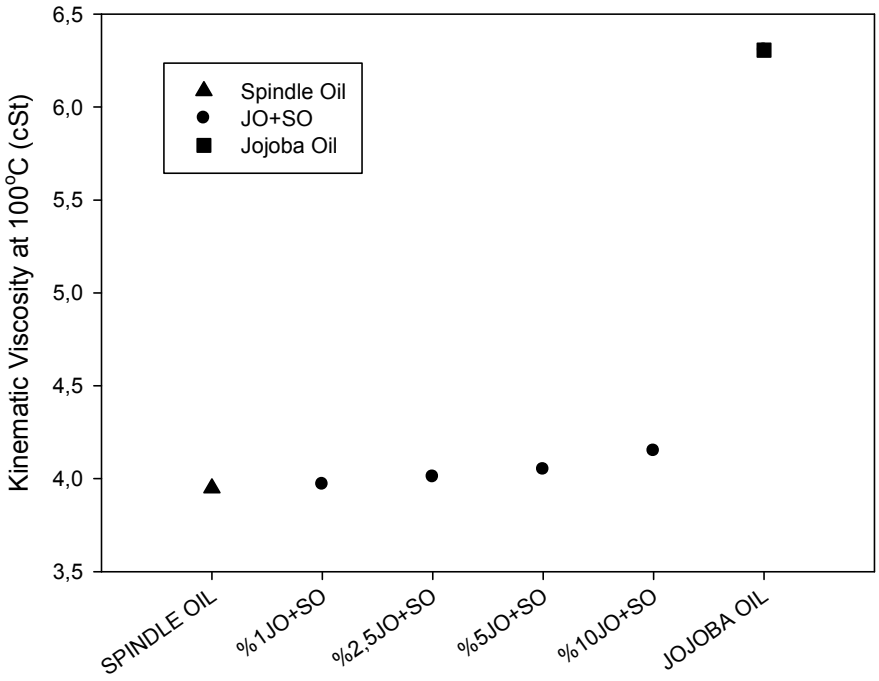


Figure 4.5. Effect of JO addition to kinematic viscosity values of SO

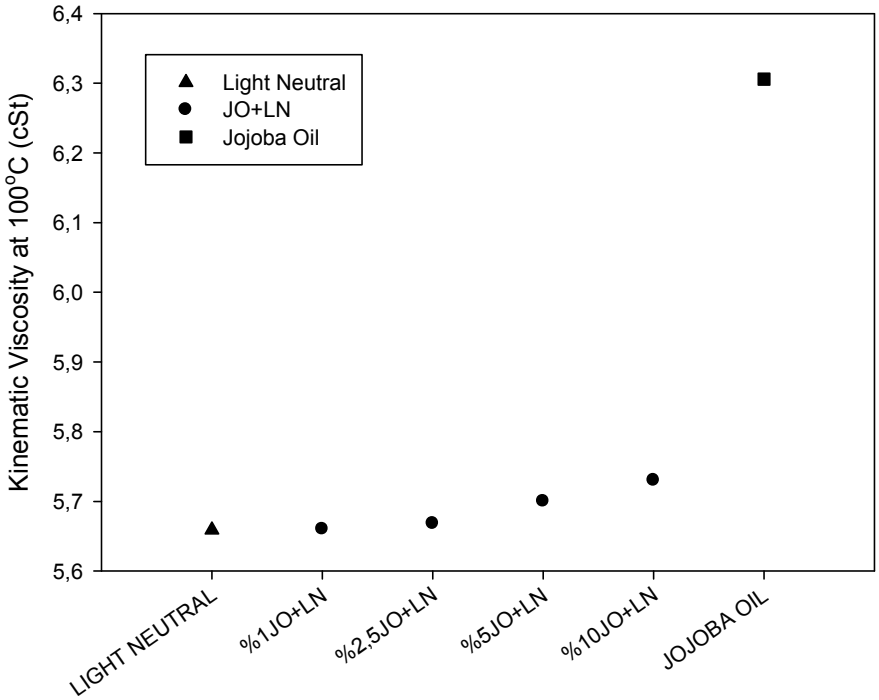


Figure 4.6. Effect of JO addition to kinematic viscosity values of LN

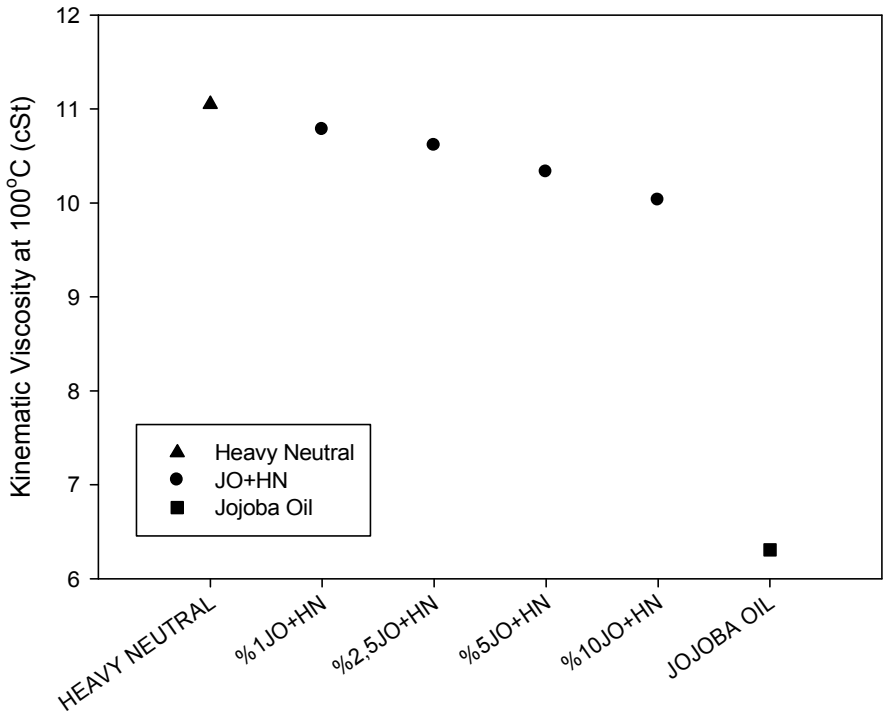


Figure 4.7. Effect of JO addition to kinematic viscosity values of HN

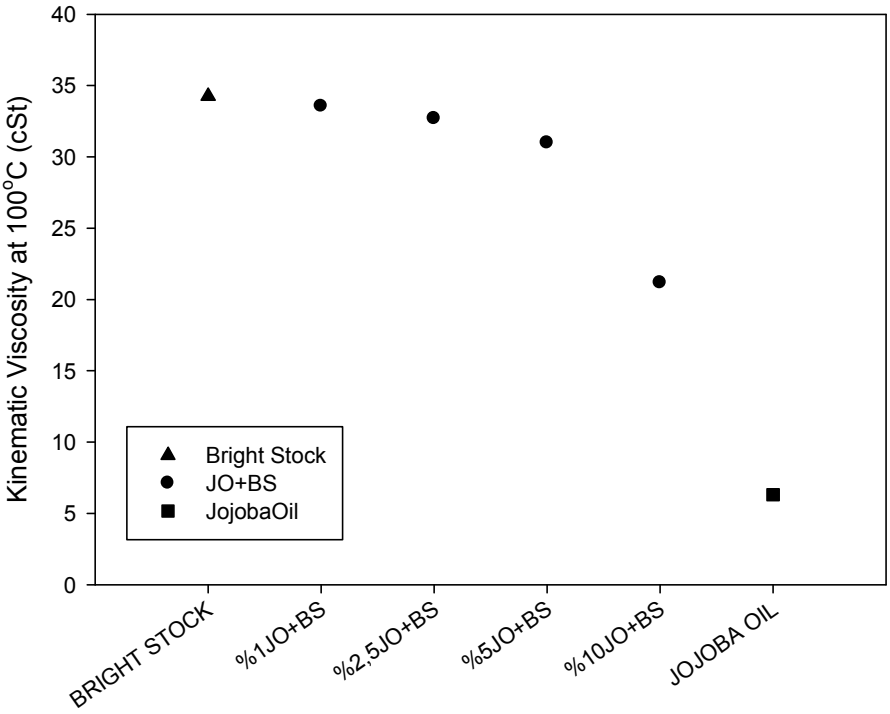


Figure 4.8. Effect of JO addition to kinematic viscosity values of BS

4.1.3. Pour Point

Petroleum base oil-derived fluids contain waxes, which at low temperatures start to crystallize to form network structures. These structures absorb oil and impede its flow (Rizvi, 2009). So it is important to measure the pour point properties of the samples. One of the major disadvantages of vegetable oils as lubricants is their high pour points. JO showed the highest pour point among the mineral base oils considered in the study. So addition of JO had to increase the pour point of the blends but the percentage of the JO in the blend was not enough to show any significant increment in the pour point of oil blends (Figure 4.9, 4.10, 4.11, 4.12). All of the pour point results were acceptable according to the standards.

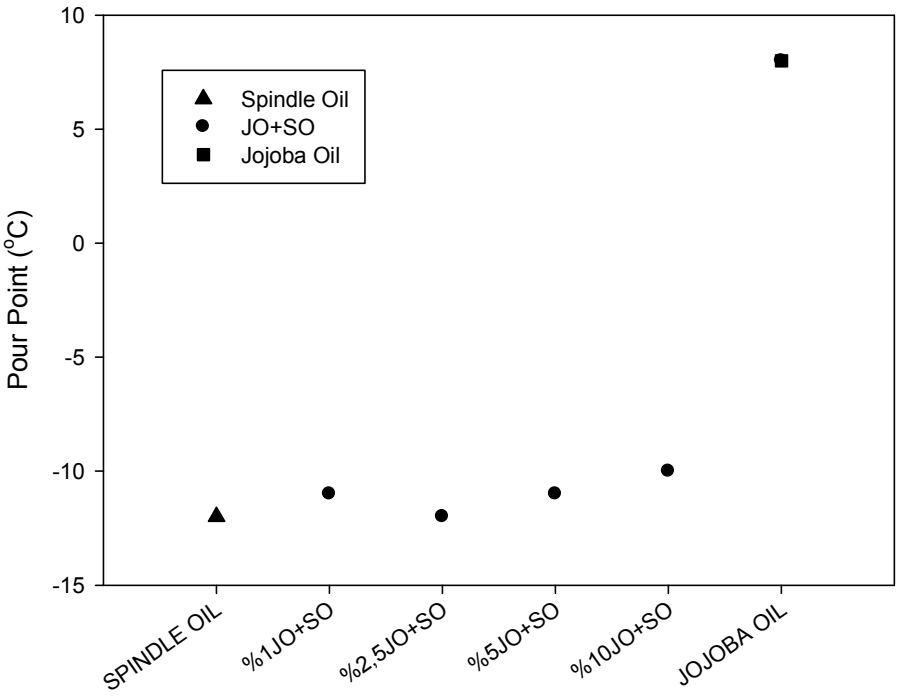


Figure 4.9. Effect of JO addition to pour point values of spindle oil

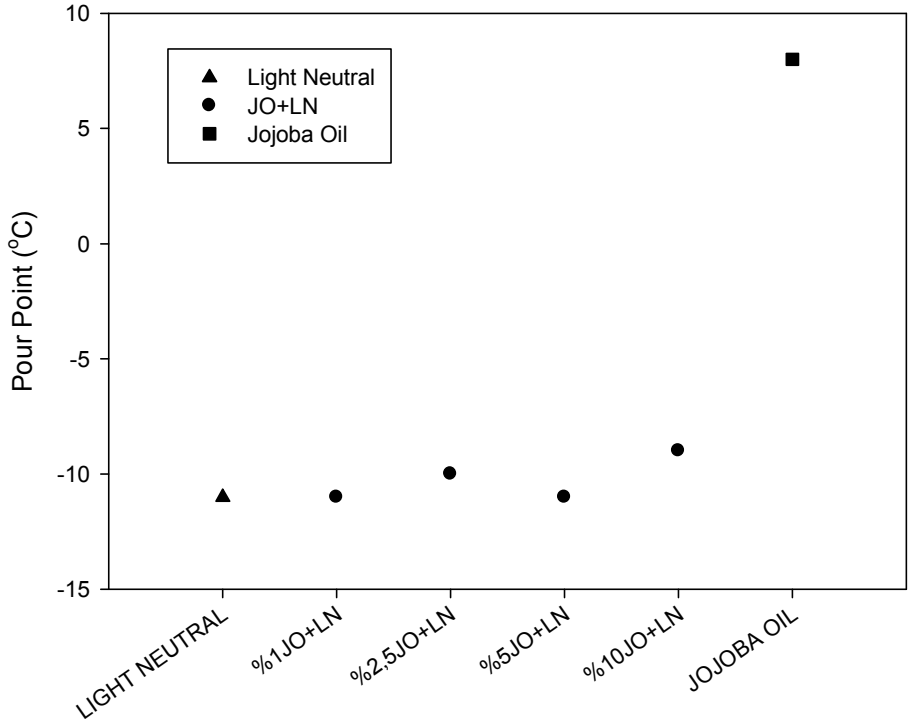


Figure 4.10. Effect of JO addition to pour point values of light neutral

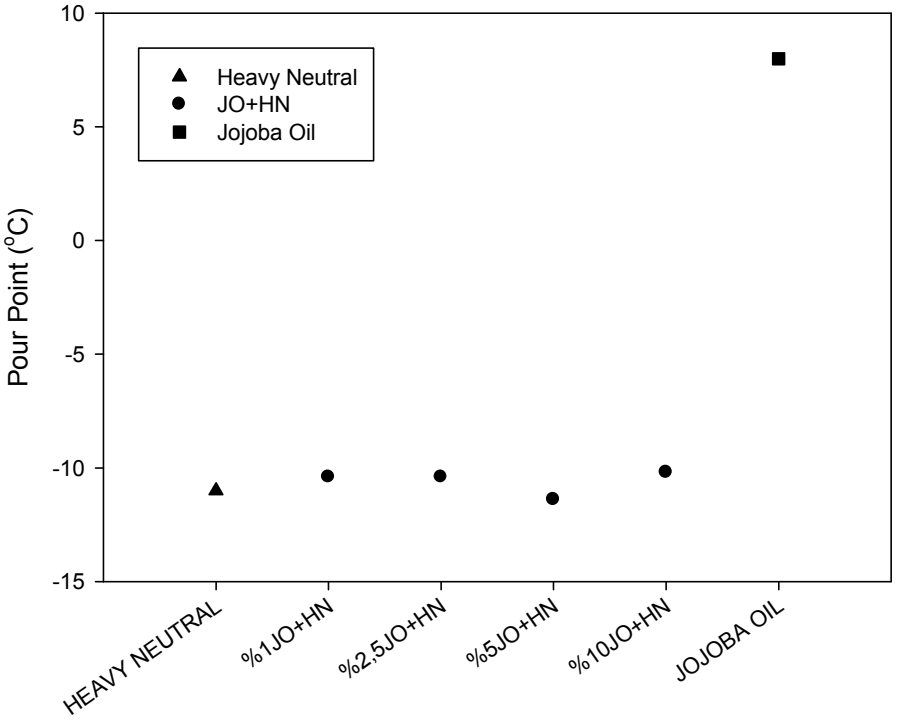


Figure 4.11. Effect of JO addition to pour point values of heavy neutral

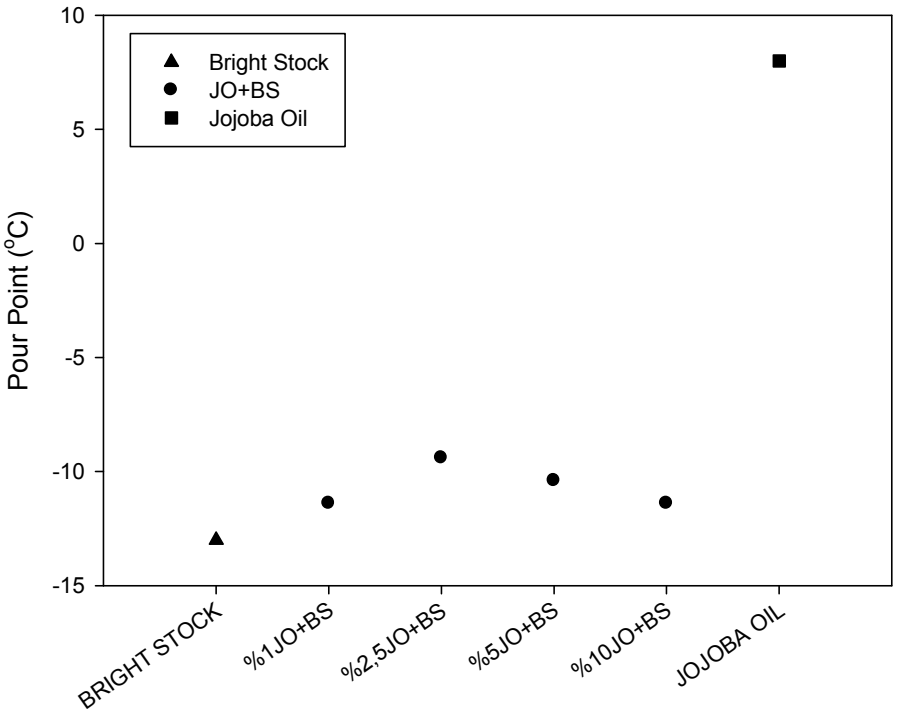


Figure 4.12. Effect of JO addition to pour point values of bright stock

4.2. Determination of Variation of the Oil Blends' Viscosity Values, Operating with Timken Test Device

Constant rate of JO additive in different kind of mineral base oil were examined in Figure 4.13, 4.14, 4.15, 4.16. Minor changes of kinematic viscosity can be seen in these figures. The reason for the minor changes among the series might be due to evaporation of the volatiles of the mineral base oil during 10 hour operation. Hence $\upsilon_{SO} \langle \upsilon_{LN} \langle \upsilon_{JO} \langle \upsilon_{HN}$ some viscosity values of the blends such as LN and SO increases while the percentage of the JO increases. On the other hand the viscosity value of the HN higher than JO because of this any increases in the percentages of JO causes a decrease in the viscosity value of the blend.

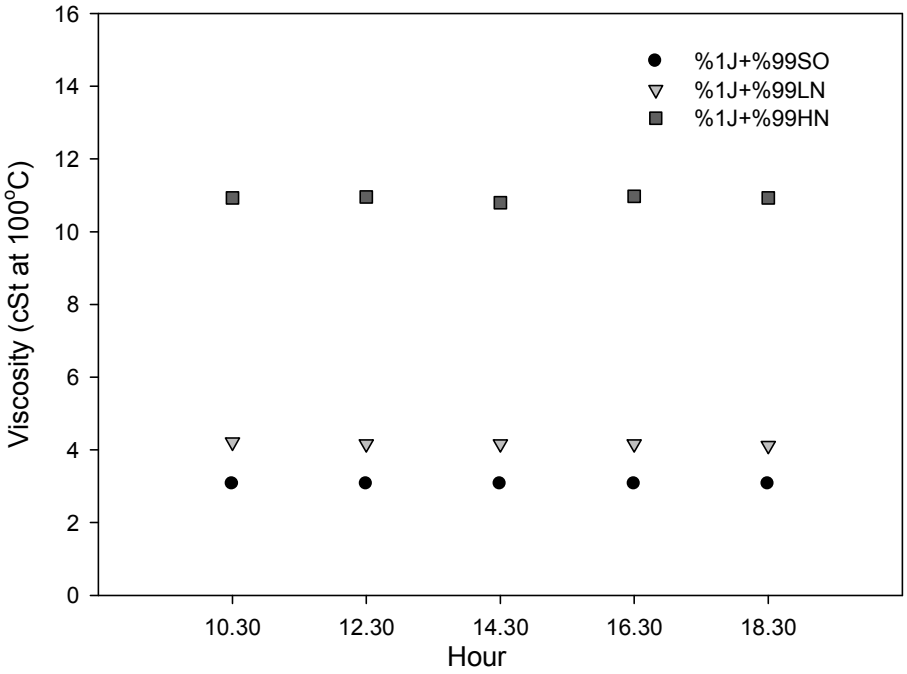


Figure 4.13 Variation of viscosity values of various blends with 1% ratio of JO

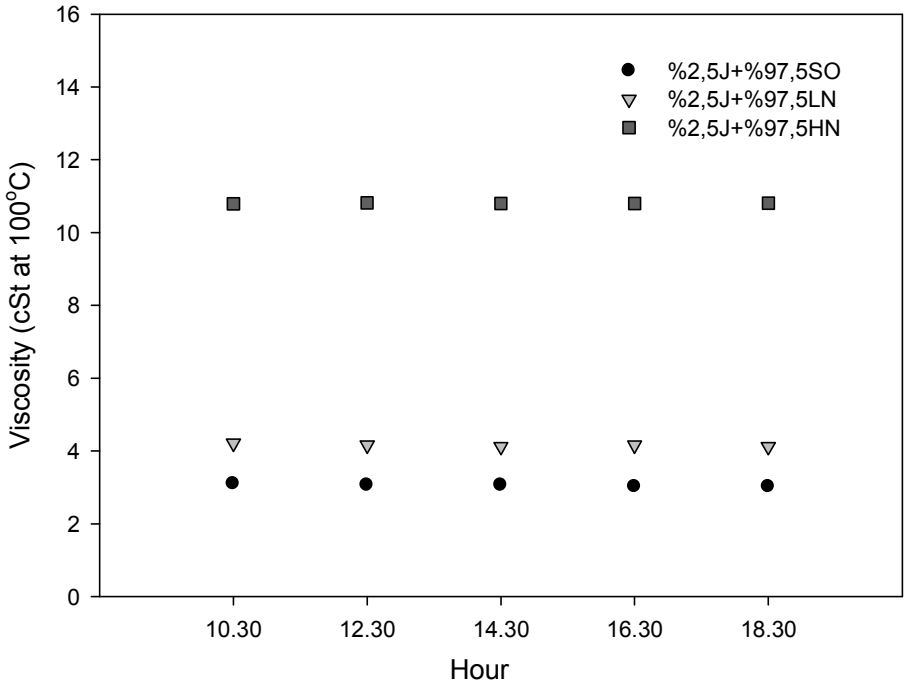


Figure 4.14 Variation of viscosity values of various blends with 2,5% ratio of JO

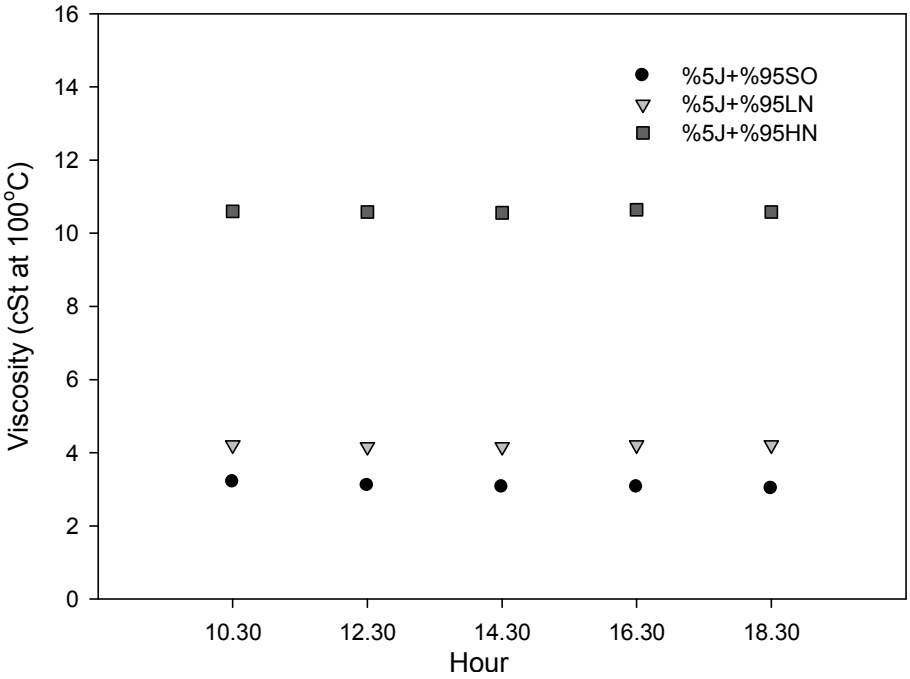


Figure 4.15 Variation of viscosity values of various blends with 5% ratio of JO

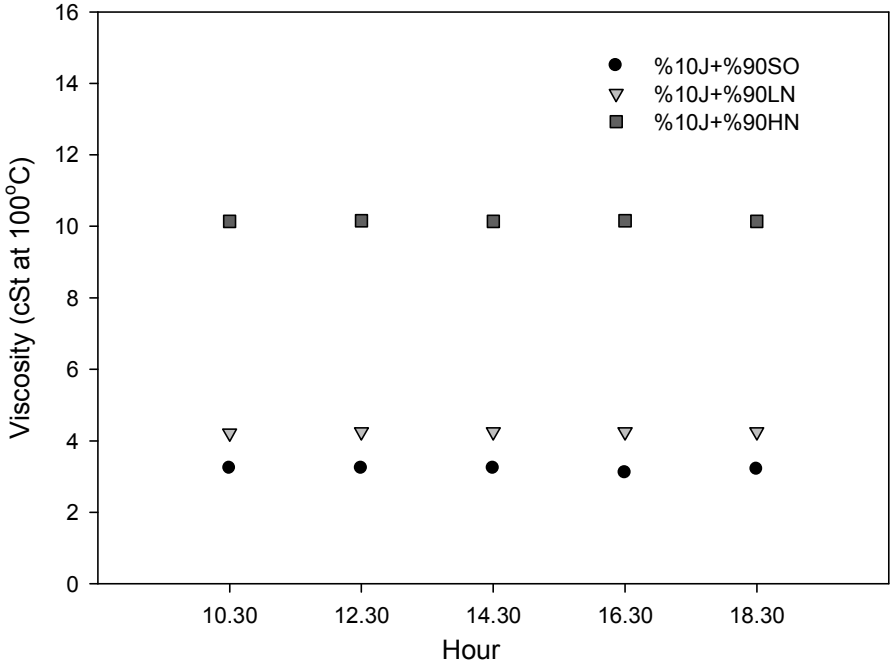


Figure 4.16 Variation of viscosity values of various blends with 10% ratio of JO

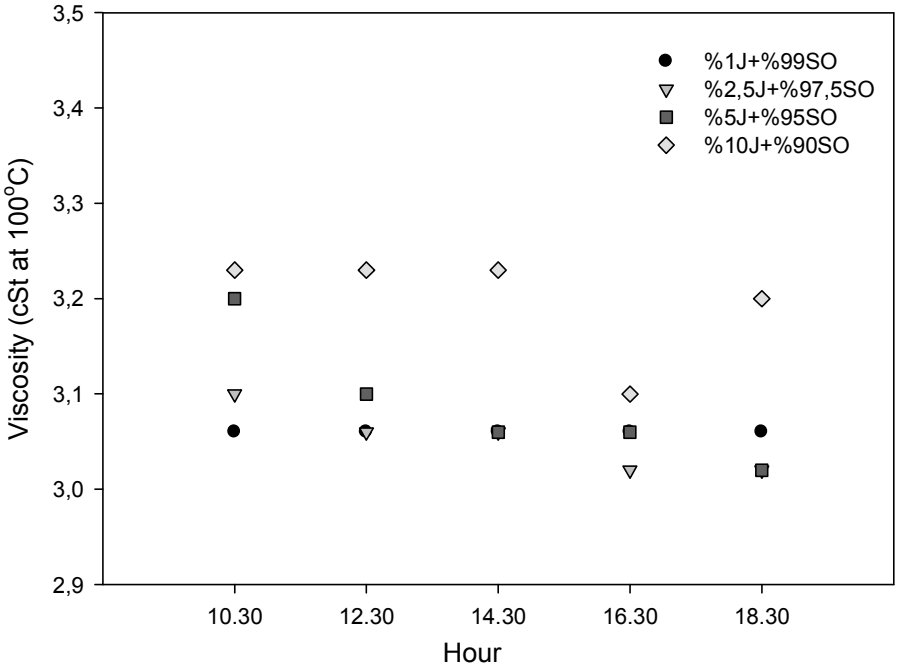


Figure 4.17 Variation of viscosity values of various ratios of JO blends in SO

Different rate of JO addition in SO were examined in Figure 4.17. Minor changes of kinematic viscosity can be seen in this figure. The reason for the minor changes among the series might be due to evaporation of the volatiles of the mineral base oil during 10 hour operation. Hence $\upsilon_{SO} \langle \upsilon_{JO}$ the viscosity values of the blends increase while the percentage of the JO increased. On the other hand the viscosity values of the SO blends show exactly the same behavior at the fourth measurement. The viscosity values of all the SO blends showed a decline then at the fifth measurement showed inclination.

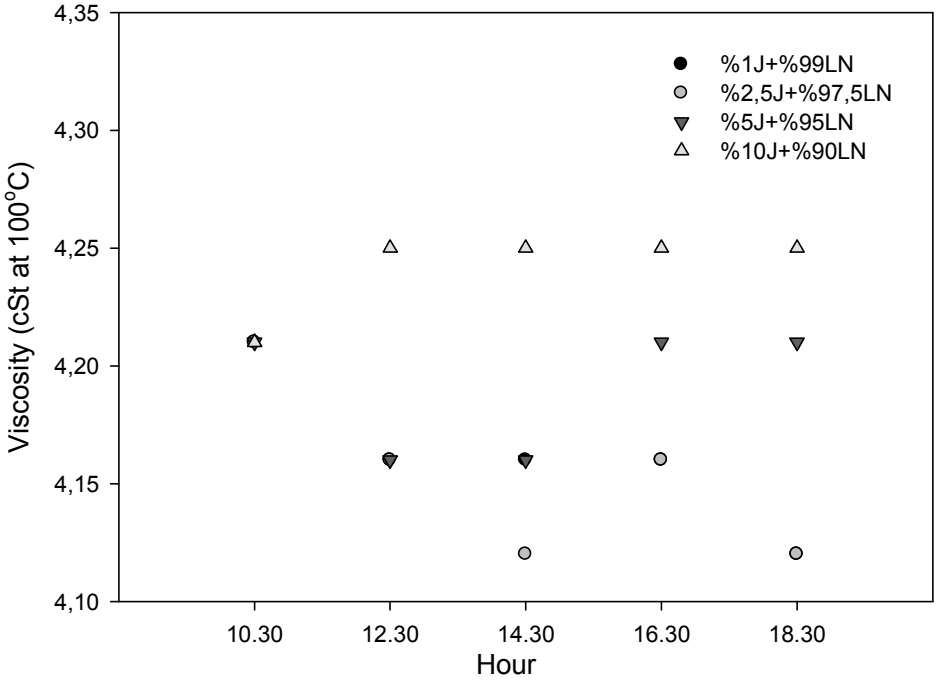


Figure 4.18 Variation of viscosity values of various ratios of JO blends in LN

Different rate of JO addition in LN were examined in Figure 4.18. Minor changes of kinematic viscosity can be seen in this figure. The reason for the minor changes among the series might be due to evaporation of the volatiles of the mineral base oil during 10 hour operation. Hence $\upsilon_{LN} \langle \upsilon_{JO}$ the viscosity values of the blends increase while the percentage of the JO increases. On the other hand the viscosity values of the LN blends show exactly the same behavior except the blend with %10 JO. The 1%, 2.5% and 5% rated blends showed decreasing behavior until third measurement (6 hours of working) however the blend with 10% JO showed the same decline at the measurement of four which means working exactly 8 hours. After this measurement blends viscosity values increased slightly.

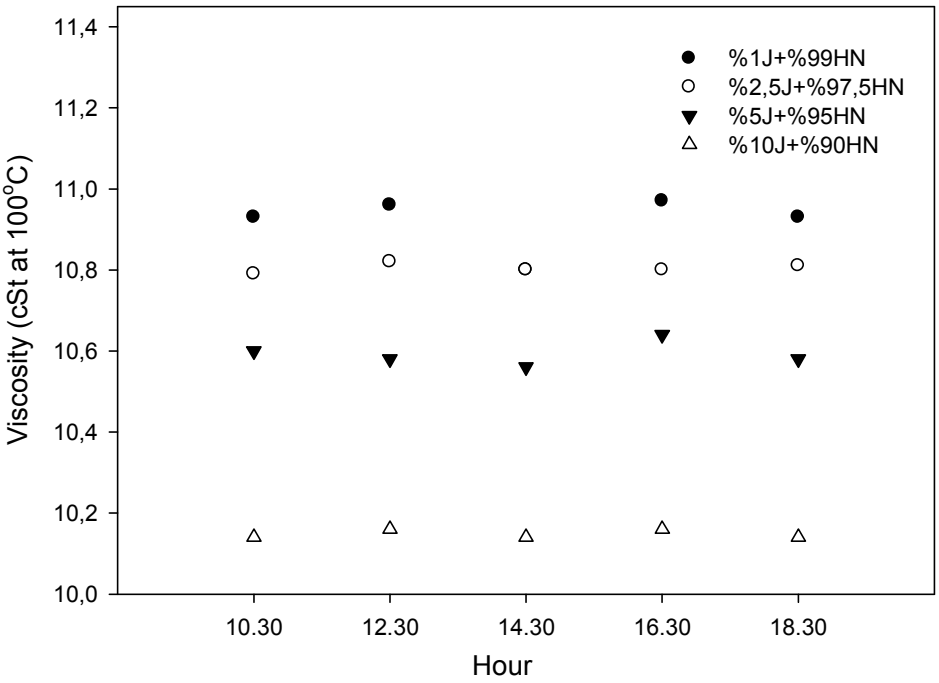


Figure 4.19 Variation of viscosity values of various ratios of JO blends in HN

Different rate of JO addition in HN were examined in Figure 4.19. Minor changes of kinematic viscosity can be seen in this figure. The reason for the minor changes among the series might be due to evaporation of the volatiles of the mineral base oil during 10 hour operation. Hence $\upsilon_{JO} \langle \upsilon_{HN}$ the viscosity values of the blends decreased while the percentage of the JO increased. On the other hand the viscosity values of the HN blends showed exactly the same behavior at the third measurement, the viscosity values of all the HN blends showed a decline then at the fourth measurement showed inclination.

5. CONCLUSION

The density of JO is lower than that of Heavy Neutral and Bright Stock, which are 0,872 g/cm^3 and 0,904 g/cm^3 respectively. Due to the lower density of JO in accordance with the Heavy Neutral and Bright Stock, blending with JO was caused a reduction in the density of the blends. Any increase in the percentage of the JO caused a decrease in the blend density. Also due to the higher density of JO in accordance with the Spindle Oil and Light Neutral, blending with JO increased the density of the blends. Any increase in the percentage of the JO caused an increase in the blend density.

The variation in the kinematic viscosity of the oil blends depends on the density values of the oil blends. JO addition to Spindle Oil and Light Neutral were caused minor increments in the oil blends beside this the addition of the oil to Heavy Neutral and Bright Stock were caused minor decrement in the oil blends. According to the higher kinematic viscosity value of the JO than the Spindle Oil and Light Neutral, kinematic viscosity of oil blends was increased.

It is important to measure the pour point properties of the samples. One of the major disadvantages of vegetable oils as lubricants is their high pour points. JO showed the highest pour point among the mineral base oils considered in the study. So addition of JO had to increase the pour point of the blends but the percentage of the JO in the blend was not enough to show any significant increment in the pour point of oil blends. All of the pour point results were acceptable according to the standards.

Constant rate of JO additive in different kind of mineral base oil were examined. Minor changes of kinematic viscosity can be seen in these figures. The reason for the minor changes among the series might be due to evaporation of the volatiles of the mineral base oil during 10 hour operation. Hence $\upsilon_{SO} \langle \upsilon_{LN} \langle \upsilon_{JO} \langle \upsilon_{HN}$ some viscosity values of the blends such as LN and SO increases while the percentage of the JO increases. On the other hand the viscosity of the HN higher than that of JO so that any increases in the percentages of JO causes a decrease in the viscosity value of the blend.

The obtained results showed that JO addition with 1%, 2,5%, 5% and 10% rates had minor effect on the lubricating properties. Therefore the use of JO at these rates is not suitable for improvement of lubricating properties. For further studies different rates (above 10%) of JO addition should be tested.

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CURRICULUM VITAE

Ayşen YILMAZ was born on February 22, 1984 in Istanbul. After completing her education in Erenköy primary and Seyhan Anatolian high school, she enrolled in Mustafa Kemal University Mechanical Engineering Department in 2003. She had graduated from the Mechanical Engineer Department of Mustafa Kemal University with a Bachelor of Science degree in 2007. She started her Master of Science education in Mechanical Engineering Department of Çukurova University in 2007. She has been working as a student at the Automotive Engineering Laboratories since 2007.

APPENDIX

Main Body

This part made on AISI 1020 steel. Technical drawing of this part shown in the Figure 1.

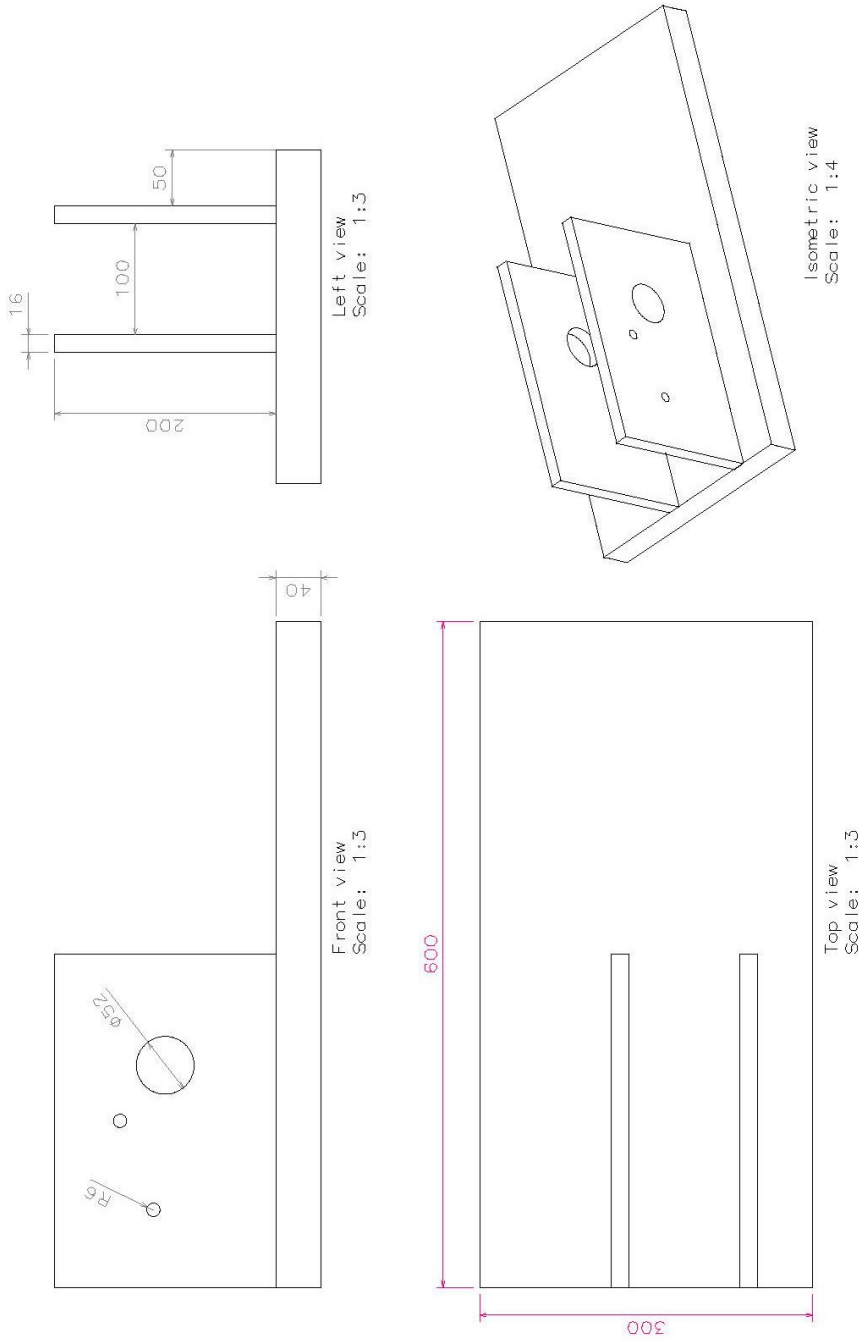


Figure 1. Technical Drawing of Main Body

Shaft

This part made on AISI 1040 steel. Technical drawing of this part shown in Figure 2.

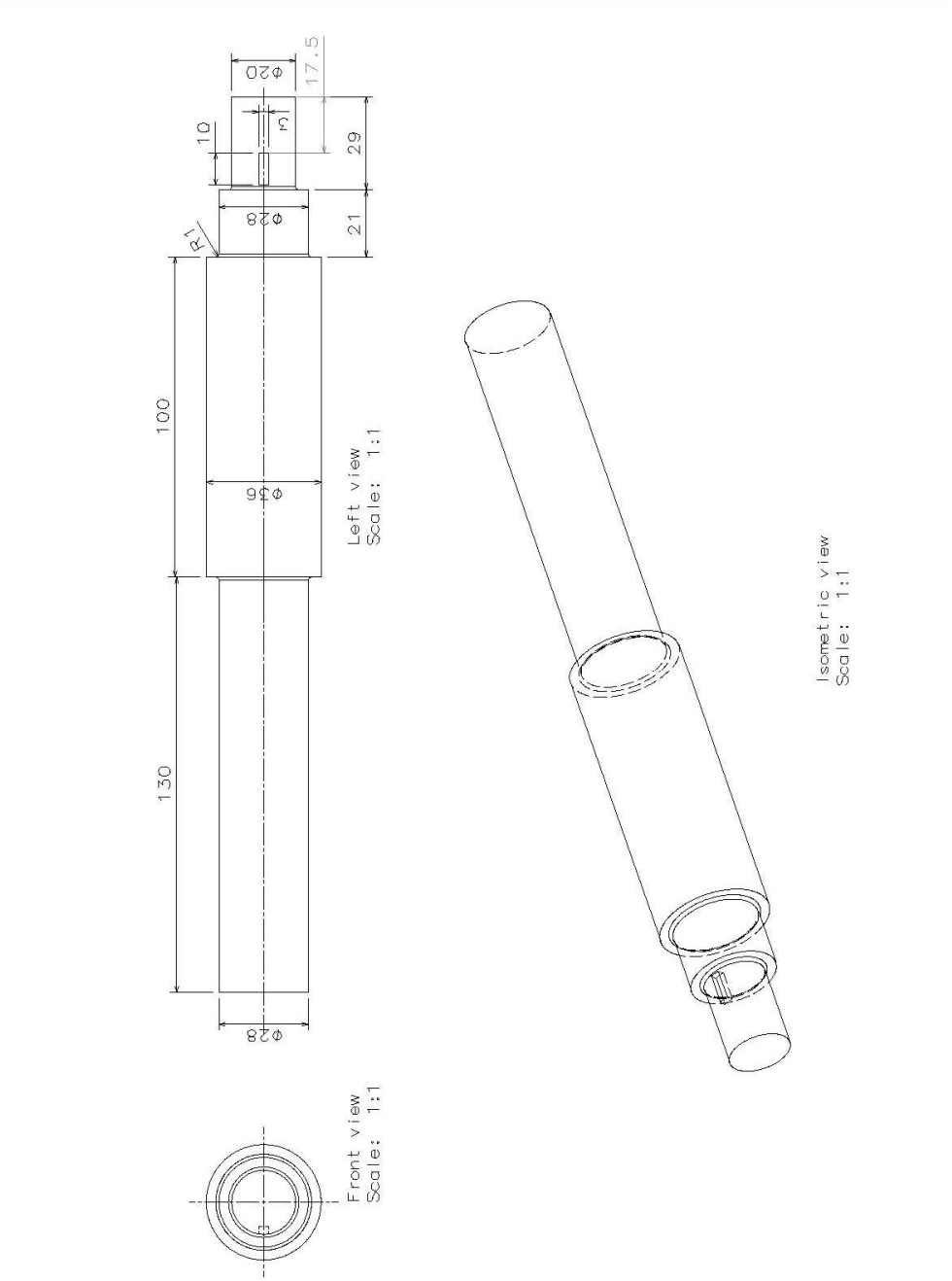


Figure 2. Technical Drawing of Shaft

Pressure Arm

This part made on AISI 1020 steel. Technical drawing of this part shown in Figure 3.

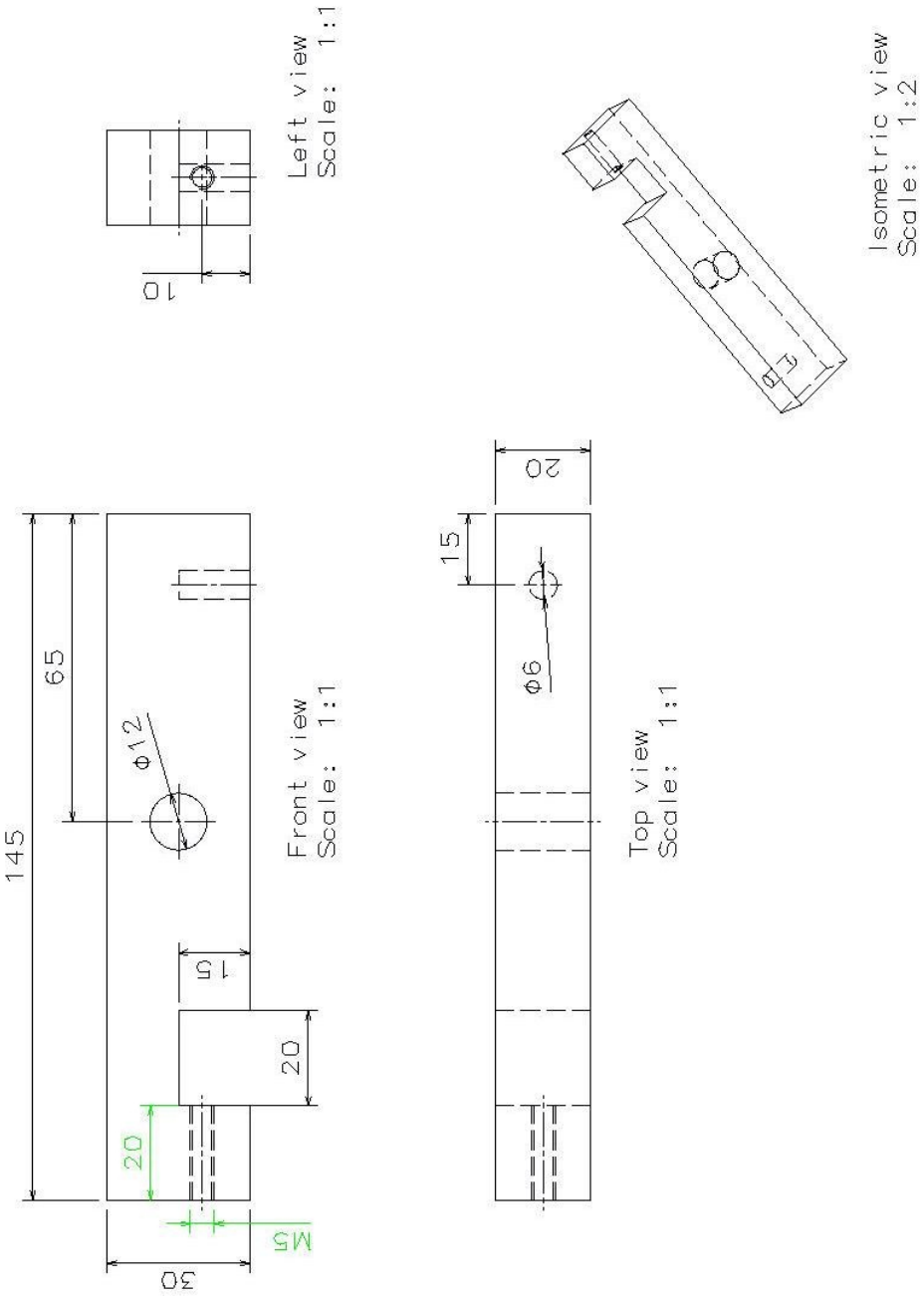


Figure 3. Technical Drawing of Pressure Arm

Weight Arm

This part made on AISI 1020 steel. Technical drawing of this part shown in Figure 4.

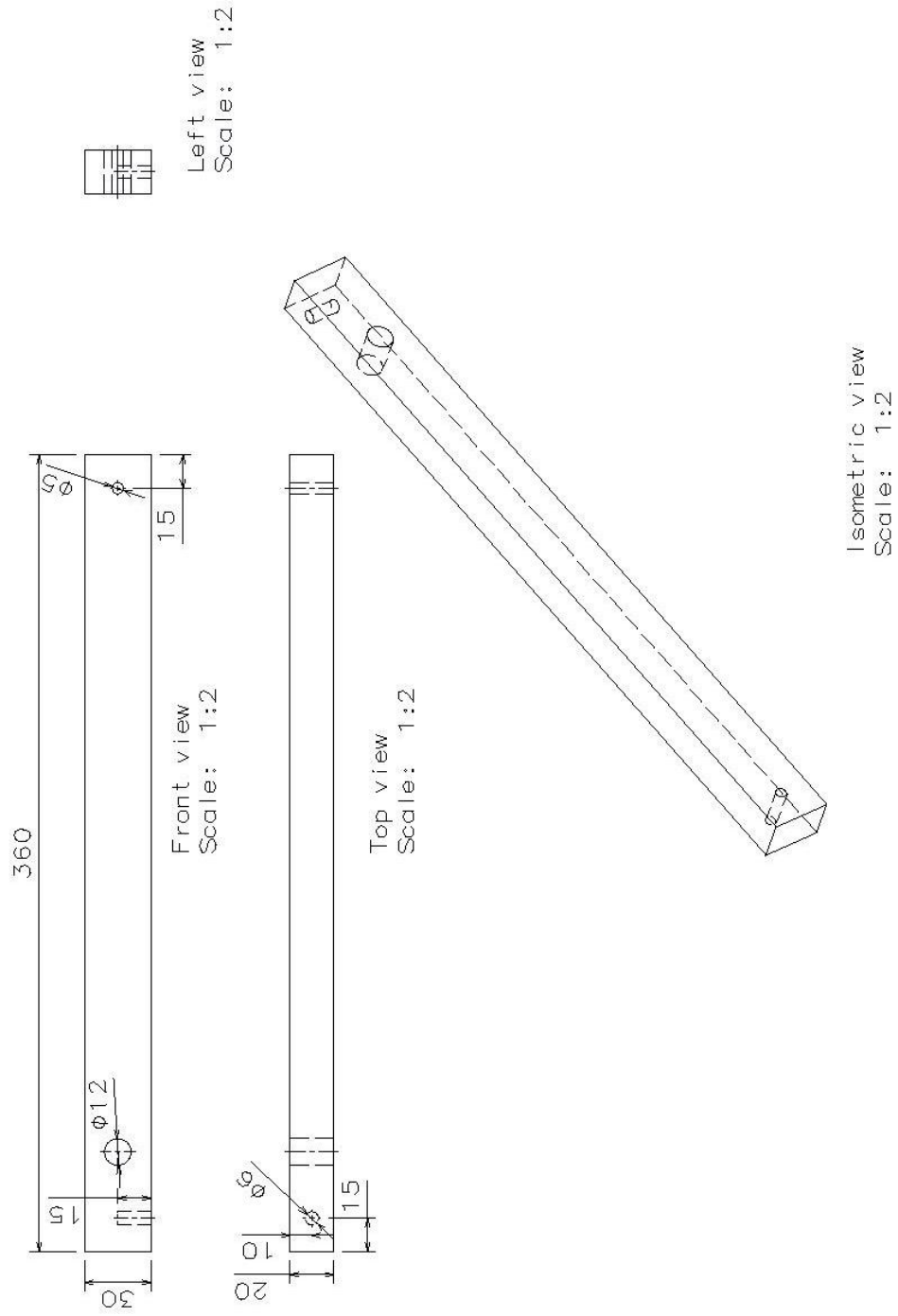


Figure 4. Technical Drawing of Weight Arm

Test Cup

This part made on 826M40 steel. Technical drawing of this part shown in Figure 5.

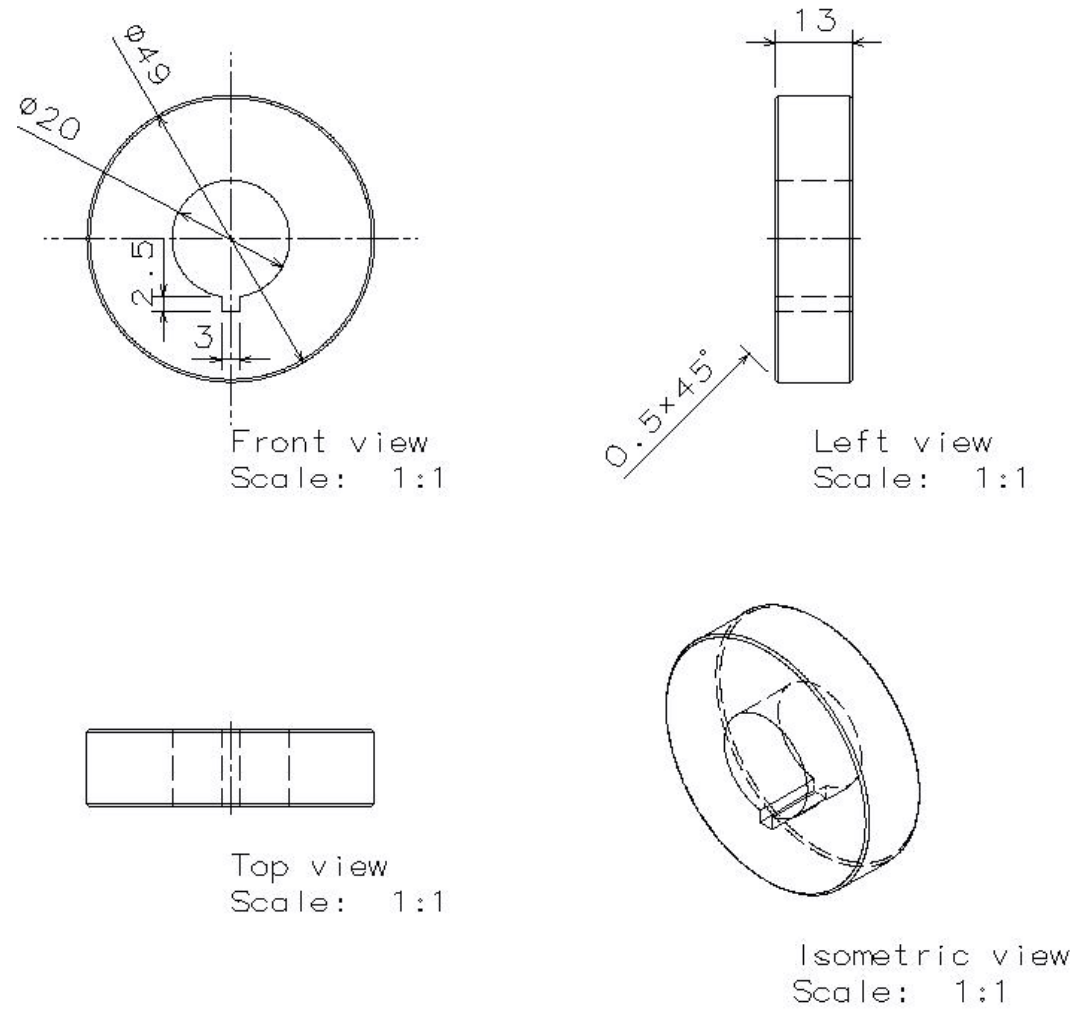


Figure 5. Technical Drawing of Test Cup