

## The Synthesis Copper-doped Nickel Oxide and Application of Hybrid Nano-lubricants as a Compressor Oil

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### ABSTRACT

Nano-lubricants have perfect heat transfer properties due to having solid nanoparticles. In this report, 2.0% copper (Cu) were doped nickel oxide (NiO) nanoparticles by a facile chemical precipitation method. The synthesized nanoparticles were physically mixed with the base liquid mineral oil (MO) at 0.5% and 1.0% mass fraction. Sodium dodecyl benzene sulphonate (SDBS) surface active material at 0.5% mass fraction was also used in the prepared suspension in order to overcome the surface tension. In this way, nano-lubricants prepared with different concentrations of NiO and Cu-doped NiO nanoparticles were used in the refrigeration system. The compressor work was calculated as 24.971 kJ/h when nano-lubricant prepared with NiO nanoparticles at 0.5% mass fraction was used in the refrigeration system. Compressor work was calculated as 23.313 kJ/h and 23.058 kJ/h when using nano-lubricant prepared with NiO nanoparticles with Cu added in 0.5% and 1.0% mass fraction, respectively, as the compressor oil. The NiO-based nanoparticles can be a promising material for high-performance hybrid nano-lubricants applications.

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## Bakır Katkılı Nikel Oksit Sentezi ve Hibrit Nano-yağlayıcıların Kompresör Yağı Olarak Uygulaması

### MAKALE BİLGİSİ

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### ÖZ

Nanoyağlayıcılar, katı nanopartiküllere sahip oldukları için mükemmel ısı transfer özelliklerine sahiptir. Bu çalışmada, %2,0 oranında bakır (Cu), basit bir kimyasal çökeltme yöntemi ile nikel oksit (NiO) nanopartiküllerine katılanmıştır. Sentezlenen nanopartiküller, ağırlıkça %0,5 ve %1,0 oranlarında baz sıvısı mineral yağ (MO) ile fiziksel olarak karıştırılmıştır. Hazırlanan süspansiyonda, yüzey geriliminin üstesinden gelmek için ağırlıkça %0,5 oranında sodyum dodesil benzen sülfanat (SDBS) yüzey aktif malzemesi kullanıldı. Bu şekilde, soğutma sisteminde farklı konsantrasyonlarda NiO ve Cu katkı NiO nanopartiküller ile hazırlanan nanoyağlayıcılar kullanılmıştır. Soğutma sisteminde ağırlıkça %0,5 oranında NiO nanopartiküller ile hazırlanan nanoyağlayıcı kullanıldığında kompresör işi 24,971 kJ/h olarak hesaplanmıştır. Kompresör yağı olarak sırasıyla; %0,5 ve %1,0 kütle oranlarında eklenmiş, Cu katkı NiO nanopartiküller ile hazırlanan nanoyağlayıcı kullanıldığında kompresör işi 23,313 kJ/h ve 23,058 kJ/h olarak hesaplanmıştır. NiO tabanlı nanopartiküller, yüksek performanslı hibrit nanoyağlayıcı uygulamaları için umut verici bir malzeme olabilir.

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## 1. INTRODUCTION (*Giriş*)

As a result of research conducted in recent years, it was predicted that non-renewable energy sources will be exhausted by the end of the 22nd century. For this reason, large budgets were allocated to the development of renewable energy sources and renewable energy systems in all world societies [1,2]. In addition to these measures for energy resources, efficient use of energy is extremely important. It is known that energy use has increased significantly in recent years throughout the world. Buildings consume more than 40% of the global energy amount and industrial systems consume the other part of the energy amount [3-5].

A significant part of the energy was used by cooling systems in industry and in our daily life. Compressors consume most of the electricity in cooling systems. In this period when we were faced with the energy crisis, efficient use of energy has become a necessity. Therefore, efficiency studies in cooling systems will be beneficial. Instead of oils used as working fluid in compressors, nano-lubricants with better heat transfer properties can be preferred. In recent studies, nano-lubricants prepared with base liquid oil and solid nanoparticles were used as compressor oil. In these prepared suspensions, solid nanoparticles in nano-lubricant are the main factor in determining the heat transfer properties [6-8].

Abbas et al. drew attention to the fact that nanofluids were used as cooling fluids in heat transfer applications. Recently, they reported that hybrid nanofluids were also used extensively in these applications. In this article, the authors have used hybrid nanofluid as the working fluid in aluminium tube radiators. They were used a new type of hybrid nanofluid containing  $\text{Fe}_2\text{O}_3\text{-TiO}_2$  (50:50) nanoparticles as the working fluid in the automotive radiator. Ultimately, it was determined that the heat transfer rate increased by 26.7% compared to water. In addition, with the new type of nanofluid used, the number of nusselt increased by 20.03% compared to water. However, a significant amount of agglomeration and sedimentation was observed when the suspension was kept for up to 20 hours after preparation [9]. One of the biggest problems in nanofluid, nano-lubricant and nano-refrigerant suspensions have seen as these agglomerations that occur in suspension after waiting for a while. Tlili et al. prepared a hybrid nanofluid by adding Ti-Cu nanoparticles into the base liquid water-ethylene. They were investigated the fluid flow and energy transport properties of the hybrid nanofluids prepared.

In the experiments, they were examined the propulsion, thermal transport, and surface friction properties of the prepared nanofluids. They were investigated numerical and graphical results for the nusselt number. It has been reported that the heat transfer rate of Ti-Cu/EG-Water hybrid nanofluid was higher than that of Ti/EG-water [10].

Sundar et al. were analyzed the entropy and exergy efficiency of nanodiamond (ND) /  $\text{Fe}_3\text{O}_4$ /60:40% W/EG hybrid nanofluids in their experimental studies. They were used the in-situ/chemical co-precipitation synthesis method while synthesizing the nanoparticles. The authors were prepared hybrid nanoparticles by adding  $\text{Fe}_3\text{O}_4$  nanoparticles to the surface of nanodiamond nanoparticles. They were predicted the magnetic properties of the prepared nanoparticles by characterization by X-ray Diffraction (XRD) and Transmission Electron Microscopy (TEM). The authors were reported that the maximum thermal conductivity performance increased up to 12.79% at  $T = 60^\circ\text{C}$ , at a nanoparticle concentration of 0.2% by volume. They were also stated that at the same concentration the nusselt number increased by 15.65% [11].

Huang et al. stated that one of the common research topics in the industry is the boiling heat transfer process. In this experimental study, a hybrid nanofluid was prepared by adding silicon oxide/graphene nanoparticles in pure water to the work nanofluid. In the experiments, the boiling heat transfer properties of hybrid nanofluids were investigated. As an effect of the study, it was noticed that the boiling heat transfer coefficient decreased [12]. Li et al. were used the hybrid nanofluid prepared in their studies as the fluid in the heat exchanger. The authors were used water as work fluid and Ag-MgO as nanoparticle while preparing nanofluid. In the experiments, the hydrothermal and irreversible properties in the forced convection flow by the heat exchanger were analysed numerically. In this study, it was stated that the heat transfer rate and the total heat transfer coefficient increased in direct proportion to the Reynolds number. It has been reported that heat exchanger efficiency, pressure loss, pumping power, and performance index are inversely proportional to Reynold's number [13]. Urmi et al. were examined the thermophysical properties of hybrid nanofluids, which they prepared in their experimental studies. They were used 40% ethylene glycol base liquid and  $\text{TiO}_2\text{-Al}_2\text{O}_3$  hybrid nanofluids while preparing nanofluid. In the experiments, they were investigated the Newtonian behavior of hybrid nanofluids depending on temperature and concentrations. The authors were

reported that the general thermal conductivity increased by 40.86% at a temperature of 80 °C and a concentration of 0.1%. In addition, it has been observed that the thermal conductivity of hybrid nanofluids is superior to single nanofluids [14].

In this study, Cu nanoparticles were added to pure NiO nanoparticles by a facile chemical precipitation method. It is known that Cu nanoparticles were frequently used in nanofluid applications and successful results were obtained [15-17]. In addition, there are experimental studies made with nanofluid prepared using NiO material [18,19]. It is seen that Cu and NiO nanoparticles were used in nanofluid applications due to their high heat transfer properties. In this experimental study, Cu-doped NiO nanoparticles synthesized by a chemical method were used to prepare hybrid nano-lubricant. [20,21]. The use of hybrid nano-lubricant were prepared with synthesized Cu-doped NiO nanoparticle in this study makes a difference. Hybrid nanoparticles are not yet common to use in nano-lubricant applications. In addition, nanoparticles were physically mixed in (50:50) ratios in general in hybrid nanofluid applications [9]. In order to prevent nanoparticle aggregation, 0.5% by weight SDBS surfactant was also added to the suspension. In the experimental study, it was aimed to show the effects of Cu-doped NiO particles synthesized by chemical methods and forming a hybrid combination on the working efficiency of the compressor in the cooling system.

## 2. MATERIAL and METHOD (MATERYAL ve METOT)

### 2.1. Experiential Procedure (Deneyisel Prosedür)

#### 2.1.1. Materials (Malzemeler)

Nickel (II) nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) (99.0%), copper (II) nitrate hydrate ( $\text{Cu}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ ) (99.99%) and sodium hydroxide ( $\text{NaOH}$ ) ( $\geq 99.0\%$ ) was obtained by Sigma Aldrich. Surfactant was used to overcome the surface tension of solid nanoparticles while preparing the suspension. Chemical structure of the used surfactant SDBS ( $\text{C}_{18}\text{H}_{29}\text{NaO}_3\text{S}$ ) was given the scheme in Fig.1 [22,23].

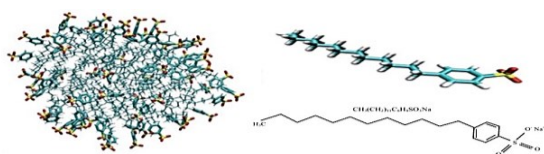


Figure 1. SDBS chemical structure. [22,23]  
(SDBS kimyasal yapısı)

### 2.1.2. Synthesis of pure NiO and Cu-doped NiO nanoparticles (Saf NiO ve Cu katkılı NiO nanopartiküllerinin sentezi)

Pure NiO nanoparticles were synthesized by a chemical precipitation method by modifying this report [24]. Firstly, 712.5 mg ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) as precursor materials was dissolved in deionized water (500 mL). After 30 min of mixing, pH degree of NiO solution was adjusted to 9.2 by 0.16 g powder of NaOH and stirred for 1 hour at room temperature. Then, obtained NiO nanoparticles were centrifuged with both water and ethanol two times at a constant rotation of 6.500 rpm. Later, obtained 40-nm sized NiO nanoparticles were dried at 80 °C for 3 h and annealed at 300 °C for 1 h. The same processes were carried out for 2% Cu-doped NiO nanoparticles. The obtained pure NiO and Cu-doped NiO nanoparticles were added in certain proportions in the oil and mixed with a homogenizer for 1 hour at room temperature.

### 2.1.3. Characterization techniques (Karakterizasyon teknikleri)

The crystallite properties of the nanoparticles were determined by Bruker D8 Advance X-ray Diffractometer (XRD). The morphologies of the pure NiO and Cu-doped NiO nanoparticles were studied using Field Emission Scanning Electron Microscopy (FESEM) (Hitachi SU5000).

## 2.2. Experimental Setup and Uncertainty Analysis (Deneyisel Kurulum ve Belirsizlik Analizi)

Stage Compressed Intermediate Cooling Training Set (Deneyset) was used in the experiments. Prepared nano-lubricants were used as operating oil in the compressor in a simple cooling system. Experimental setup was shown in Fig. 2.

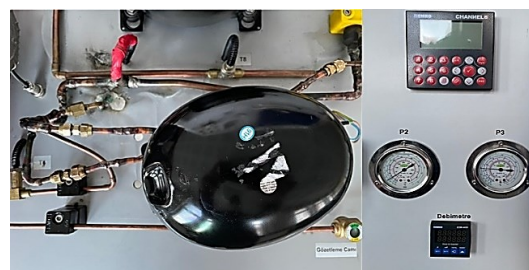


Figure 2. Image of experimental setup.  
(Deneyisel kurulumun resmi)

Pt-100 type thermocouples operating in the range of - 200 °C ~ +850 °C were used for temperature measurements in the experiment set. In the experiments, Refco Type (mr-205,305-ds) oil manometers were used to measure the pressure values.

According to the Holman [25] model, the uncertainties of the measurement elements were calculated as  $\pm 1.41$  and  $\pm 0.10$ , respectively.

### 2.3. Preparation of Nano-lubricant (Nano-yağlayıcının hazırlanması)

When preparing nano-lubricant, 100 ml of MO was used as the base fluid. Solid-liquid suspension was obtained by adding NiO and Cu-doped NiO into mineral oil in different mass fractions. In the first stage, in the suspension, 0.5% by weight NiO was used as the nanoparticle. In the next step, Cu-doped NiO nanoparticles were used in 0.5% and 1.0% mass fractions by weight. In order to prevent flocculations in the suspensions, 0.5% by weight SDBS surfactant was added. Solid-liquid mixture was mixed with Ultrasonic homogenizer to ensure a homogeneous distribution in the suspension. Ultrasonic homogenizer is a good tool for even distribution of solid particles in the liquid. Then, the prepared nano-lubricants were mixed in an ultrasonic water bath (Kudos-Model: SK2210HP) for 3 hours. Finally, the suspension was also stirred in magnetic stirrer (Jeio Tech MS-32M) for 3 hours. These stages were shown in the Fig. 3.

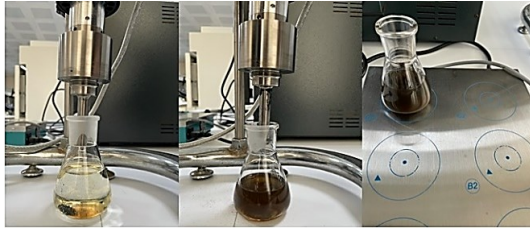


Figure 3. Preparation of different concentrations of nano-lubricants. (Farklı nano yağlayıcı konsantrasyonlarının hazırlanması)

## 3. RESULTS and DISCUSSION (SONUÇLAR ve TARTIŞMA)

### 3.1. XRD and SEM Results for Cu-doped NiO Nanoparticles (Cu katkılı NiO Nanopartikülleri için XRD ve SEM Sonuçları)

To reveal the crystallite properties of the synthesized pure NiO and Cu-doped NiO nanoparticles, XRD analysis were carried out, as shown in Fig. 4. The 37.37°, 43.38°, 62.85°, 75.26 and 79.39° characteristic peaks for (111), (200), (220), (311) and (222) orientations of the pure NiO powder in XRD pattern were shown in Fig. 4a. Peaks from XRD are well matched with primary status of 00-047-1049 JCPDS card number of pure NiO. The well matched with the related JCPDS card indicated that NiO was high pure [26,27] On the other hand, in the case of 2.0% Cu incorporation into NiO structure, the

peak intensity of Cu-doped NiO powder was raised, which indicates the improvement of the crystallite properties [28,29]. In addition to crystallite improvement, in the case of 2.0% Cu incorporation into NiO structure, the position of the corresponding peak was shifted to up to  $\sim 0.4^\circ$  higher points, as shown in Fig. 4b. The position shift of Cu-doped NiO sample reveals that the Cu ions was successfully incorporated into NiO structure and which may mentioned improvements a lots of properties the pure NiO sample [30]. Moreover, the average crystallite size of obtained pure NiO and Cu-doped NiO nanoparticles was calculated from the Debye-Scherrer equation [31-34]. The calculated average crystallite size for pure NiO was approximately 4.57 nm for dominant peak (the position of 43.37°), while average crystallite size for pure Cu-doped NiO was approximately 7.34 nm for dominant peak (the position of 43.76°).

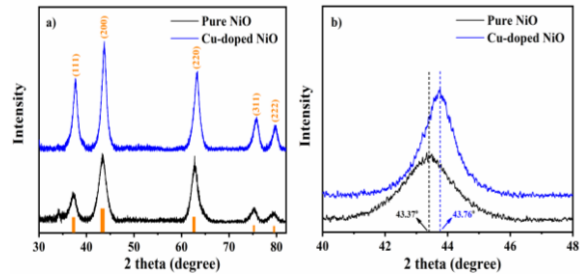


Figure 4. (a) XRD pattern of pure NiO and Cu-doped NiO. (b) XRD shift of pure NiO and Cu-doped NiO at 43.37° position. (a) Saf NiO ve Cu katkılı NiO'nun XRD modeli b) 43.37° konumunda saf NiO ve Cu katkılı NiO'nun XRD kayması)

To observe the morphologies properties of the pure NiO and Cu-doped NiO nanoparticles, SEM measurements were made, as shown in Fig. 5. Fig. 5a shows the pure NiO SEM images while the Fig. 5b shows Cu-doped NiO SEM images. As seen in Fig. 5, the grains of both nanoparticles were dispersed without clustering. Comparing surface images of nanoparticles, the surface images of produced nanoparticles have been not changed significantly with Cu doping effect. On the other hand, the grain size in both samples is below 50 nm, which is an important parameter for oil performance. In addition to SEM images, energy dispersive X-ray (EDX)-mapping method was used to detect the distribution of Cu ions in the structure of NiO host materials. Obtained EDX-mapping results of Cu and Ni ions showed in Fig. 5(c-d). As can be seen from the EDX-mapping results in Fig. 5d, Cu ions are distributed homogeneously in the structure of NiO.



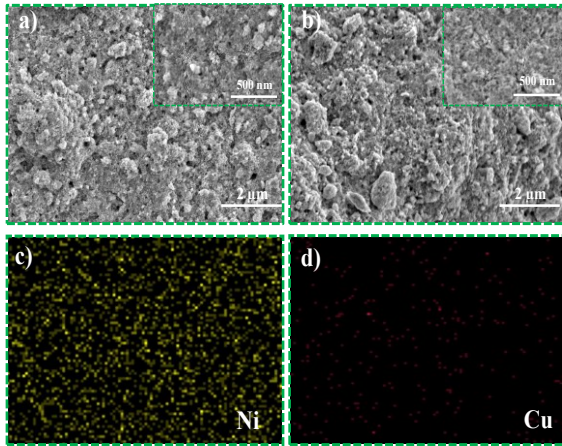


Figure 5. SEM images of a) pure, b) Cu-doped NiO powders at different magnification. (*Saf ve Cu katkılı NiO tozlarının SEM görüntüleri*). EDX-mapping images of c) Ni, d) Cu in synthesized Cu-doped NiO powders. (*Sentezlenmiş Cu-katkılı NiO tozu için EDX-haritalama sonucu*)

### 3.2. Investigation of Compressor Capacity and Coefficient of Performance (*Kompresör Kapasitesi ve Soğutma Etkinlik Katsayısının İncelenmesi*)

In this paper, different concentrations of nano-lubricants have been prepared with base fluid oil, hybrid nanoparticles and surfactant material. These prepared nano-lubricants were used as compressor oil in the cooling cycle and the thermodynamic parameters for the cycle were evaluated. In the cooling cycle; the inlet-outlet temperatures of the compressor, evaporator and condenser elements and the pressure values of the system were measured. Based on these measurement results, enthalpy values were determined thermodynamically. Depending on the temperature, pressure and flow rate of the refrigerant, the compressor capacity, the amount of heat the evaporator absorbs from the ambient and the COP values were calculated based on the following formulas [35].

$$\text{COP} = \frac{\dot{Q}_E}{\dot{W}_C} \quad (1)$$

$$\dot{W}_C = \dot{m}(h_2 - h_1) \quad (2)$$

$$\dot{Q}_E = \dot{m}(h_1 - h_4) \quad (3)$$

Condenser outlet and evaporator inlet enthalpy values were averaged while calculations were made. Also, the performance of cooling systems was affected by the ambient temperature. While conducting the experiments, the ambient temperature was measured between 14 and 16 °C. In the experiments, firstly, pure MO was utilized as the operating fluid in the compressor. Then, experiments

were carried out with nano-lubricants prepared with different concentrations of NiO nanoparticles and hybrid Cu-doped NiO nanoparticles. In line with the data obtained as a result of the experiments, the compressor work and coefficient of performance were calculated as given in Fig. 6. When pure MO was utilized as the compressor operating oil, the compressor duty was calculated as 26.836 kJ/h, while the coefficient of performance was calculated as 4.66. Then, when the nano-lubricant prepared with NiO nanoparticles at 0.5% ratio by weight was utilized as compressor oil, the compressor capacity was calculated as 24.971 kJ/h, and the coefficient of performance was calculated as 5.09. When hybrid Cu-doped NiO nanoparticles were used while preparing the nano-lubricant, the compressor work was calculated as 23.313 kJ/h and the coefficient of performance was calculated as 5.45. When we compare the use of hybrid nano-lubricant with the use of pure MO oil, the compressor work has decreased by 13.12%.

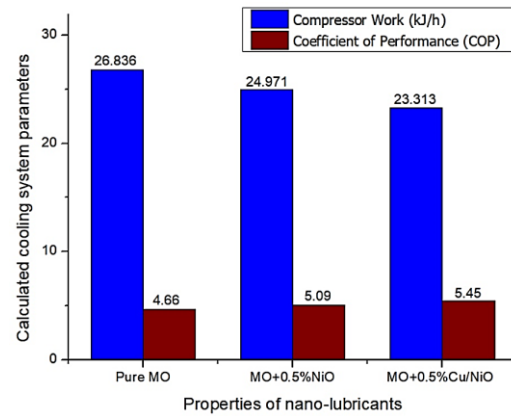


Figure 6. Compressor work and COP values (MO/NiO/Cu-doped NiO) (*Kompresör çalışması ve COP değerleri (MO/NiO/Cu katkılı NiO)*).

The effects of using hybrid nano-lubricants in different mass fractions in the vapor compression cooling system were also shown in Fig. 7. As a conclusion of the use of pure MO as compressor work fluid, the compressor capacity was calculated as 26.836 kJ/h and the coefficient of performance was calculated as 4.66. When the mass fraction of Cu-doped NiO nanoparticles used in preparing hybrid nano-lubricants was made 1.0%, the compressor work was calculated as 23.058 kJ/h and the coefficient of performance was calculated as 5.52. When we compare the use of hybrid nano-lubricants at 1.0% mass fraction with the use of pure MO, the compressor work has decreased by 14.07%. Precipitation was observed when the suspension was prepared with

more than 1.0% by weight of nanoparticles while preparing the nano-lubricant.

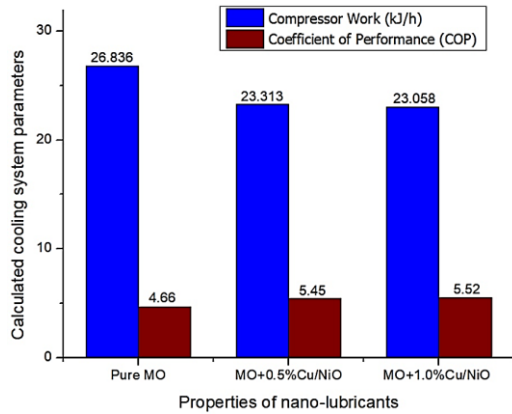


Figure 7. Compressor work and COP values (MO and different mass fraction Cu doped NiO)  
(Kompresör çalışması ve COP değerleri (MO ve farklı kütle fraksiyonu Cu katkılı NiO))

The performance of the cooling system depends on the ambient conditions. However, a comparison has been made with a previous study. Akkaya et al. in their experimental study, a different type of compressor was used. The COP value was calculated as 4.70 when nano-lubricant prepared with base liquid polyol ester (POE) and 1.0% Titanium dioxide ( $\text{TiO}_2$ ) was used [7]. In this study, the highest COP value was calculated as 5.52. However, the ambient conditions differed in this study. In addition, different types of compressors and nano-lubricants were used.

#### 4. CONCLUSIONS (SONUÇLAR)

In this study, NiO nanoparticles were synthesized and then Cu nanoparticles were added by a facile chemical precipitation method. While synthesizing the nanoparticles, 2.0% by weight of Cu nanoparticles in NiO were used. Hybrid nanoparticles synthesized were tested in the cooling system. While preparing the nano-lubricant, nanoparticles in 0.5% and 1.0% mass fractions and surface active material in 0.5% mass fraction were used. Studies have been carried out with pure MO oil and different concentrations of nano-lubricants and the experimental results have been compared.

- When SEM and XRD results were examined, it is seen that the hybrid nanoparticles synthesized are synthesized as pure and homogeneously. The presence of Cu particles in NiO nanoparticles is seen in these analyses.
- All nano-lubricants different proportions by weight operated the refrigeration machine more efficiently than pure MO.

- When pure MO was utilized as compressor work fluid, the compressor capacity was calculated as 26.836 kJ/h. When utilized as nano-lubricant compressor oil prepared with base liquid MO and 1.0% Cu doped NiO, the compressor work was calculated as 23.058 kJ/h. When using hybrid nano-lubricant as compressor oil, compressor work has been reduced by approximately 14.07%.
- When pure MO was used in the compressor, the COP value was calculated as 4.66. COP rate was calculated as 5.52 when used as nano-lubricant compressor work fluid prepared with base liquid MO and 1.0% Cu-doped NiO. When the hybrid nano-lubricant was utilized as the compressor work fluid, the COP value has increased by 18.45%.
- In the experiments, it has been concluded that hybrid nano-lubricants can be used safely in the cooling system. It can also be suggested that hybrid nano-lubricants with different properties can be used.

#### ABBREVIATIONS

$\dot{Q}_E$	The amount of heat absorbed by the evaporator (kJ/h)
$\dot{W}_C$	Compressor work (kJ/h)
$\dot{m}$	Mass flow rate (kg/h)
$h_{1,2,4}$	Enthalpy values (kJ/kg)
T	Temperature [K]
Cu	Copper
NiO	Nickel oxide
Ag	Silver
MgO	Magnesium oxide
$\text{Ti}_2\text{O}$	Titanium dioxide
$\text{Al}_2\text{O}_3$	Alumina oxide
ND	Nanodiamond
MO	Mineral oil
SDBS	Sodium dodecyl benzene sulphonate
COP	Coefficient of performance
XRD	X-Ray diffraction
FESEM	Field emission scanning electron microscopy
TEM	Transmission electron microscopy

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