Improvement of Physicochemical Properties of Light-Exposed Linseed Oil by Blending with Nanoparticles

Areej Ahmed Yassin [1], Amira Fawzy Soliman [2,*], Naglaa Mohammed Abd Al-Fattah [3]

[1] Lecture nutrition and food science, Clinical nutrition department, National Hepatology and Tropical medicine research Institute, Egypt.

[2] Lecturer of Biochemistry, National Heart Institute, Giza, Egypt

[3] Lecturer of Nutrition, Growth and Nutrient Requirements Department, National Nutrition Institute (NNI), Egypt

*** Corresponding author, Email address: *amira_fawzy2013@yahoo.com***

ABSTRACT

It's interesting to learn that silver nanoparticles (Ag NPs) not only have an antibacterial effect but also provide a good level of safety in fields where people eat and touch things. It has received food and drug administration (FDA) certification in the United States and is nontoxic, odorless, and harmless. Future possibilities for these nanomaterials offer a variety of opportunities for the creation of novel goods and uses in the food system. Linseed oil may have a longer shelf life if Ag NPs are added as preservatives in different doses. The sample was examined by transmission electron microscopy and UV spectroscopy after being pyrolyzed into Ag NPs in oleic acid. Through storage times, assessments of rancidity and microbiological characteristics were conducted. The findings demonstrated that while shelf life rises compared with the control sample, Ag NPs decrease total bacterial count and chemical properties, except iodine value. This technique can completely alter how food is produced. The prospects for these nanomaterials offer numerous opportunities for the creation of novel goods and uses in the food chain.

Keywords: *Silver Nanoparticles, Linseed Oil, rancidity.*

INTRODUCTION

"Many, many rules had begun to bend at the hand of nanotechnology, gene therapy, robotics, artificial intelligence. This produced a lot of good and a lot of bad. This trade-off has always plagued us. When you make waves, you produce peaks and troughs". Nanotechnology has recently been shown to have the ability to improve the agri-food sector, reducing unfavorable human health concerns, agricultural practices' impact on the environment, and enhancing food productivity and security, all while promoting social and economic fairness **(Mukhopadhyay, 2014; Wani et al., 2020). Cruz-Lopes et al., (2021)** concluded that nanoscience and nanotechnology are the twenty-first century's new horizons. The ability to alter materials on a nanoscale is referred to as nanotechnology. Recent advancements in the fields of nanoscience and nanotechnology, like those in other industries, have opened up new avenues for food innovation. Nano foods are foods that have been cultivated, produced, processed, or packed utilizing nanotechnological technologies to improve nutritional quality, flavor, or texture, as well as extend the shelf life of the product.

Linseed oil (LO, Linum usitatissimum, Flaxseed) is one of the richest plant sources of α-linolenic acid (ALA), ω-3 ALA, which has been linked to a variety of health benefits **(Joshi et al., 2022).** In many follow-up studies, High ALA consumption has been shown to reduce the risk of fatal myocardial infarction **(Calling et al., 2019).** In addition, ALA-rich vegetable oils can help to protect the heart and circulatory system **(Nguyen et al., 2018). Rodríguez-Correa and others (2020)** noted that ALA intake enhanced vascular function, reduced inflammation, and caused bodyweight loss in patients who were overweight to obese and had metabolic syndrome symptoms. Like many other vegetable oils, extended exposure to sunshine can cause lipid oxidation in LO. When exposed to food, light is a photochemical initiator that can cause photochemical reactions **(Zeb and workmates, 2008).** Vegetable oil photochemical oxidation's mechanism has been well investigated **(Choe and Min, 2006; Koutchma, 2019),** and the oxidation of lipids in the presence of light is known as photo-oxidation. Because it contains a lot of unsaturated fatty acids, LO is vulnerable to oxidation. Additionally, studies have shown that antioxidants that stop oil from oxidizing might be damaged by prolonged UV exposure **(Oh et al., 2014).** Peroxides and other volatile and toxic chemicals are created by photo-oxidation in oils. As a result, the oil is less stable and has less economical, nutritional market value as well as less safety. Importantly, the oils lose their flavor and sensory quality, become unappealing to customers, and become undesirable, all of which cause financial losses to the food and nonfood industries **(Redondo-Cuevas and colleagues, 2018).**

The aim of the present work is to use silver nanoparticles (AgNPs) as preservatives of LO, to improve the quality of the oil.

MATERIALS and METHODS

Materials

Sigma-Aldrich supplied analytical quality Silver Nitrate ($AgNO_3$), trisodium citrate ($Na_3C_6H_5O_7$), oleic acid, potassium iodide (KI), sodium thiosulphate ($Na_2S_2O_3$), starch, acetic acid (CH_3COOH), and chloroform. Linseed oil (LO) was purchased from the local market randomly. All the water was distilled and then purified to Millipore Milli-Q standards. Before use, all glassware was washed in a bath of newly prepared aqua regia solution (HCl/HNO_3, 3:1) and then thoroughly rinsed with H_2O.

Instruments

In quartz cuvettes, the colored solution was measured using a Perkin Elmer Lambda-40 spectrophotometer. Transmission electron microscope (TEM) experiments were carried out utilizing a JEOL-JEM 1200 electron microscope with a 90 kV accelerating voltage. A drop of solution containing the particles was put on a copper grid covered with amorphous carbon for the TEM observations. The excess solution was removed with blotting paper after allowing the film to stand for 2 minutes, and the grid was allowed to dry before the measurement. The diameters of the particles were assessed using micrographs taken with a Joel-100S transmission electron microscope at 120 kV.

Methods

Preparation of silver nanoparticles (Ag NPs)

Ag NPs were made by reducing Ag (I) to Ag (0) with trisodium citrate as the reducing agent and oleic acid as the stabilizing agent, as described below **(Liu and Lu, 2006; Turkevich and associates 1951):**

10 ml of 5 mM (AgNO3) was heated to boil at 200 °C then; 2 mL 1% trisodium citrate was added drop by drop to 100 ml of oleic acid. After 5 minutes of heating, the solution was withdrawn from the heater and swirled for another 15 minutes. The reaction solution's color was seen to change from colorless to yellow, then turbid. Finally, there was no further change, suggesting that the reaction was complete.

Chemical analysis

PV was calculated using the approved method Cd 8b-90 **(AOCS, 2004).** AV was calculated using the Ca5a-40 modified official technique **(AOCS, 2004).** Official method 920.158 was used to calculate IV **(AOAC, 2005).** P-anV was determined using the official technique Cd 18-90 **(AOCS, 2004),** and the value of TBA was determined using the official method Cd 19-90 **(AOCS, 2004).**

Microbiological analysis

Oxoid provided nutrient agar, a microbiological growth medium extensively used for regular bacterial cultivation. According to **Aneja (2010),** a total bacterial count was performed.

RESULTS and DISCUSSION

Preparation and Characterization of Silver Nanoparticles (AgNPs)

Umadevi et al (2012) declared that when the frequency of the electromagnetic field becomes resonant with the coherent electron motion, a significant absorption is produced, which is the beginning point for the seen color, whose absorption is highly dependent on particle size, dielectric medium, and environmental conditions. The UV-Vis absorption spectrum of silver nitrate, Ag (NO3), solutions (Figure 1a) indicates the formation of AgNPs in the visible range of 426 nm, whereas the absorption peaks of silver nitrate, Ag (NO3), solutions would appear at 310 nm due to the presence of nitrate ions in the solution **(Nakamura et al., 2011)**. Figure 1b showed the TEM morphological shape of Ag NPs was mainly spherical with an average diameter of 8-15 nm. The findings of this study support prior research, indicating that the surface plasmon resonance (SPR) peak for spherical Ag NPs occurs between 410 and 480 nm **(Krutyakov et al., 2008; Kuntyi et al., 2019; Rahman and others, 2018; Sharifi-Rad et al., 2021).**

Effect of silver nanoparticles (Ag NPs) on linseed oil quality characteristics

The shelf-life quality and consequently the economic value of oils are determined by several quality control parameters, including iodine value (degree of unsaturation), peroxide value (formation of primary oxidation products), P-anisidine value, thiobarbituric acid, and acid value (formation of free fatty acids due to rancidity) **(Decker et al., 2010; Endo, 2018).** Storage without and with silver nanoparticles (Ag NPs) at varying concentrations of 17, 3.4, and 0.34 ppm, under varied storage conditions (dark and light), and for three storage durations allowed for the analysis of the quality features (zero time, after 2, and four months): IV, TBA, PV, P-anV, and AV.

Peroxide value (PV)

Daily diet includes a significant amount of vegetable oils. It's important to note that because peroxide levels increase during storage, all vegetable oils become more rancid. These peroxides may strengthen the radical that fractures lipids, resulting in turbidity and a disagreeable flavor and odor. The peroxide value (PV) (meq/kg) of linseed oil (LO) while being stored in the light and the dark with and without Ag NPs is shown in Figs. 2a and 2b. The figure demonstrated that the peroxide value (PV) value of LO was (3.2 ± 0.06 meq/kg) at zero time in the absence of Ag NPs and that it increased with an increase in storage time (4 months), reaching 11.3 ± 0.17 meq/kg (Fig. 2a). While the peroxide value (PV) of samples devoid of silver nanoparticles exposed to light was higher than that of samples subjected to darkness over the same time (32.4 ± 1.3 meq/ kg) (Fig. 2b). The photo-degradation of the oil fats and the subsequent production of additional free radicals may be the cause of the peroxide value increasing in the presence of light. These findings are consistent with previous publications **(Anwar et al., 2007; Mikołajczak & Tańska 2022)** that indicated a higher rate of primary oxidation product generation in oils exposed to light. The figure also demonstrated that the peroxide value dropped in the presence of Ag NPs under the same storage circumstances (dark and light) compared to the control sample and that the decline was dependent on the quantities of the silver nanoparticles in the oil. The presence of Ag NPs in the dark, as depicted in Fig. 2a, resulted in the lowest peroxide value of LO, followed by Ag NPs in the light (Fig. 2b). Additionally, it was shown that, when compared to samples without nanoparticles stored under the same conditions, the lowest concentration of 0.34 ppm of Ag NPs had the minimum PV in both dark and light, followed by concentrations of 3.4 ppm and 17 ppm. The presence of nanoparticles is predicted to function as a reflector, scatter the majority of visible and near-infrared light, operate as an electron scavenger, and reduce the peroxide value.

P-anisidine value (P-anV)

The P-anisidine value (P-anV) of linseed oil was displayed in Figures 3a and 3b after dark and light storage with and without silver nanoparticles. P-anV values were determined to be 9, 10.3, and 13.5 at 0, 2, and 4 months, respectively, for all samples that were stored in the dark without the presence of silver nanoparticles. At the same intervals, there was light present at 16.1, 19.8, and 24.9, respectively. The increase in P-anisidine value could be attributed to the high rate of secondary oxidation product synthesis, which accelerated oxidative deterioration **(Ismail et al., 2016; Shahidi & Wanasundara, 2002).** All samples' P-anV values were lower than those of the control samples when Ag NPs were present during dark and light storage. The highest effective concentration of silver nanoparticles in both light and darkness was 0.34 ppm, as shown in Figures 3a and 3b. The presence of Ag NPs is anticipated to inhibit oxidative stress processes and function as a free radical scavenger. The P-anV is a trustworthy indicator of oxidative rancidity in fats, oils, and fatty meals, according to **Yılmaz Tuncel and Yılmaz Korkmaz (2021).**

Iodine value (IV)

A crucial feature of oils is IV, which indicates how much of the oil's unsaturated fatty acids (double bonds) are present **(Kumar et al., 2012).** This quantity reflects the oil's unsaturated fatty acids before and after oxidation. Therefore, IV is a valuable metric for analyzing changes in the unsaturated fatty acid levels in LO exposed to sunlight since it reveals the oxidative stability of the oil **(Dodoo and workmates, 2021).**

Figures (4a&4b) depicted the relative change in the iodine value (IV) of linseed oil (LO) that was stored in the dark and light for four months while also being treated with NPs. These figures (4a&4b) demonstrated that oil samples exposed to light exhibited a greater rate of IV degradations than ones held in darkness. These findings were in line with those of **Dodoo et al. (2021); Naz et al (2005)** who observed that oil samples stored in light experienced a greater fall in iodine values than oil samples stored in darkness. Due to the increased rate of photo-oxidative degradation, this drop may be explained by the greater loss of unsaturated fatty acids. The iodine value (IV) is regarded as a significant indicator of the deterioration of oils since it is characterized by a decline in the total unsaturated contents of the oil. It establishes the resistance of oils to oxidation and enables the qualitative determination of the total unsaturation of the fat **(AOCS, 2004; Maliki & Ifijen, 2020).** Although at a slower rate than that of their counterparts without Ag NPs, the IV of samples containing Ag NPs fell. The samples with concentrations of 0.34 ppm of Ag NPs in both dark and light had the highest IV, as shown in Figures 4a and 4b.

Thiobarbituric Acid (TBA)

Figures 5a, and b depicted the relative change in the TBA value of the LO stored in the dark, light, with, and without Ag NPs for four months. The findings shown in figures (5a & 5b) showed that when storage time was extended, TBA increased for all oil samples. Additionally, these findings showed that the TBA of oil samples stored in light was higher than that of similar samples maintained in darkness. These findings supported those of **Al-Dalain et al (2011); Cheng and workmates (2019)**, who found that the growth of TBA values was linearly associated with storage time and exposure to light. Malondialdehyde (MDA), a minor part of fatty acids with three or more double bonds, is created during lipid oxidation as a result of the hydrolysis of polyunsaturated fatty acids. Both for the early appearance when oxidation occurs and for the sensitivity of the analytical approach, it serves as a pointer to the lipid oxidation process **(Cesa, 2004; Rasul, 2021).** Figure 5a and 5b show that the TBA of samples containing silver nanoparticles (Ag NPs) was lower than that of control and that the TBA of samples declined as Ag NPs concentrations reduced to 17 ppm, 3.4 ppm, and 0.34 ppm.

Acid value (AV)

The acid value (AV) (mg/g) of LO held in darkness and light, with and without silver nanoparticles, was shown in Figures 6a& 6b. The graph showed that all oils' acid values increased as storage times increased **(Gulla and Waghray, 2011; Maszewska et al., 2018**), and samples exposed to light had higher acid values than those kept in the dark **(Anwar et al., 2007; Koyuncu & Tunçtürk, 2017).** Additionally, it was noted that the acid value of the oil samples containing Ag NPs was lower than that of the control samples. Figure 6a of the same figure showed that samples containing 0.34 ppm Ag NPs had the lowest acid value in darkness, whereas samples containing 0.36 ppm Ag NPs had the lowest acid value in light.

Microbiological analysis (total bacterial counts)

During the lengthy time of storage, it was shown that samples containing silver nanoparticles had fewer bacteria than samples containing no such particles. The total bacterial count of linseed oil was shown in Table 1 for both light and dark storage conditions, with and without silver nanoparticles. The table showed that samples containing silver nanoparticles had fewer microbes than ones containing no nanoparticles. These findings supported those of **Al-Nori (2012); Ansari et al. (2021),** who revealed that gold and silver nanoparticles have a great deal of antibacterial ability for eradicating both gram-positive and gram-negative bacteria.

Additionally, it was shown that the number of bacteria increased with prolonged storage. These findings corroborated **Zhou's (1999)** finding that the number of microorganisms in vegetable oils increased during simulated storage. It was determined that when the concentration of silver nanoparticles fell, so did the number of bacteria.

It's remarkable to note that even oil samples without increases in microbes had varying degrees of increases in AV, PV, TBA, and P-anV following storage. These findings corroborated those of **Boran et al (2006); Zhou (1999)** who claimed that non-biological variables were to blame. Furthermore, all samples IV dropped during storage. **Boran et al (2006)** noted that this was caused by oxidative and hydrolytic rancidity, which raised AV and lowered IV. Oil samples with microbes had a higher acid value. Increases in the acid value had a positive impact on the number of microorganisms. **Montaño López et al (2022) and Zhou (1999)** explained that this was because the microorganisms could break down fats into free fatty acids (FFA), which would increase the value of the microorganisms and possibly favor their metabolism.

With lower PV, PanV, TBA, AV, and lower rates of decreasing their IV, samples with NPs may have had higher oxidative stability than those without NPs. This may also be because gold and silver nanoparticles were excellent antibacterial agents **(Ismail et al., 2014)**

This suggested that LO would degrade more quickly during storage without the addition of Ag NPs. In clusters up to several microns in size, primary NPS frequently congregate **(Méndez et al., 2020).** For Ag NPs in the dark, the lowest concentration ($0.34X10^{-3}$ ppm) was the most effective. These findings were in agreement with those of **Cordray and workmates (2012),** who noted that higher concentrations increased the speed at which samples aggregated. They were also in agreement with **Duan et al (2011) and Ma et al. (2020),** who noted that higher concentrations increased the likelihood of collisions, which led to more nanoparticle aggregation.

While the lowest concentration of Ag NPs was the most efficient concentration in light this agreed with **Cheng et al. (2019); Iqbal et al (2021)** who noted that under sunlight irradiation, nanoparticles irreversibly aggregated with different degrees. The UV content of the sunlight is specified to be the driving force of nanoparticle aggregation, and the strong oscillating dipole-dipole interaction is believed to be the origin of the destabilization.

CONCLUSIONS

It may be simple and quick to transition from the lab to grocery store shelves and our kitchen tables when using silver nanoparticles to enhance the quality of linseed oil (LO) and other fatty oils. This technique can completely alter how food is produced. The prospects for these nanomaterials offer numerous opportunities for the creation of novel goods and uses in the food chain.

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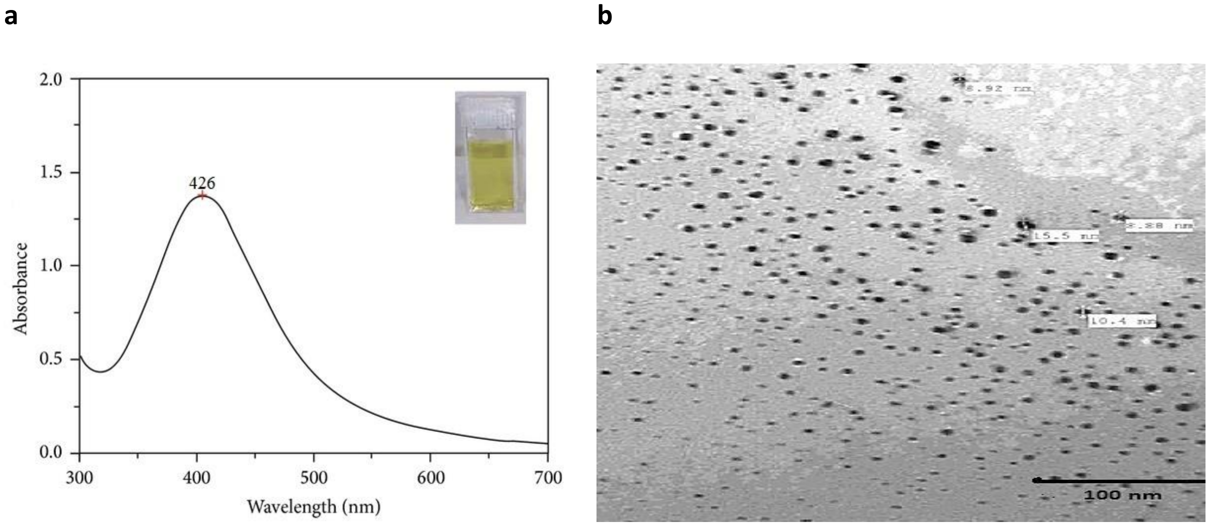

Figure (1): (a) UV-Vis absorption spectrum of Ag NPs in oleic acid, (b) TEM image of Ag NPs

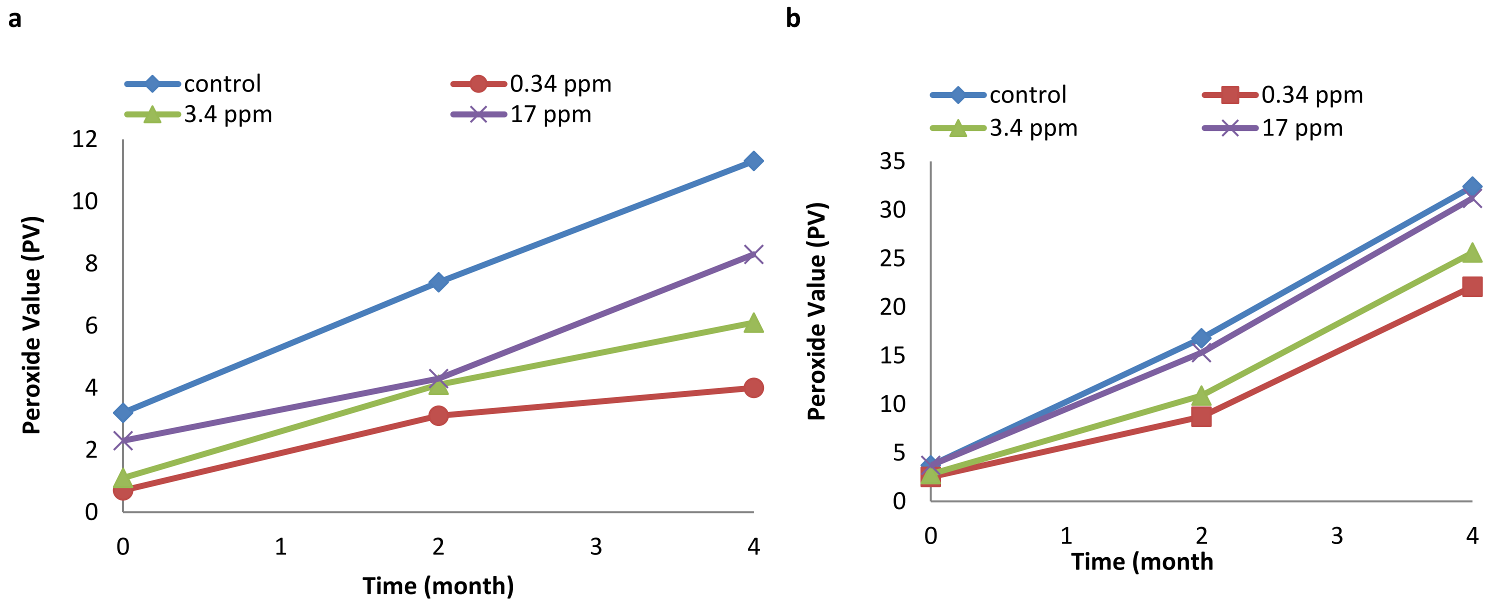

Figure (2): PV (meq/Kg) of Linseed oil (LO) with and without Ag NPs during storage in (a) dark, (b) Light

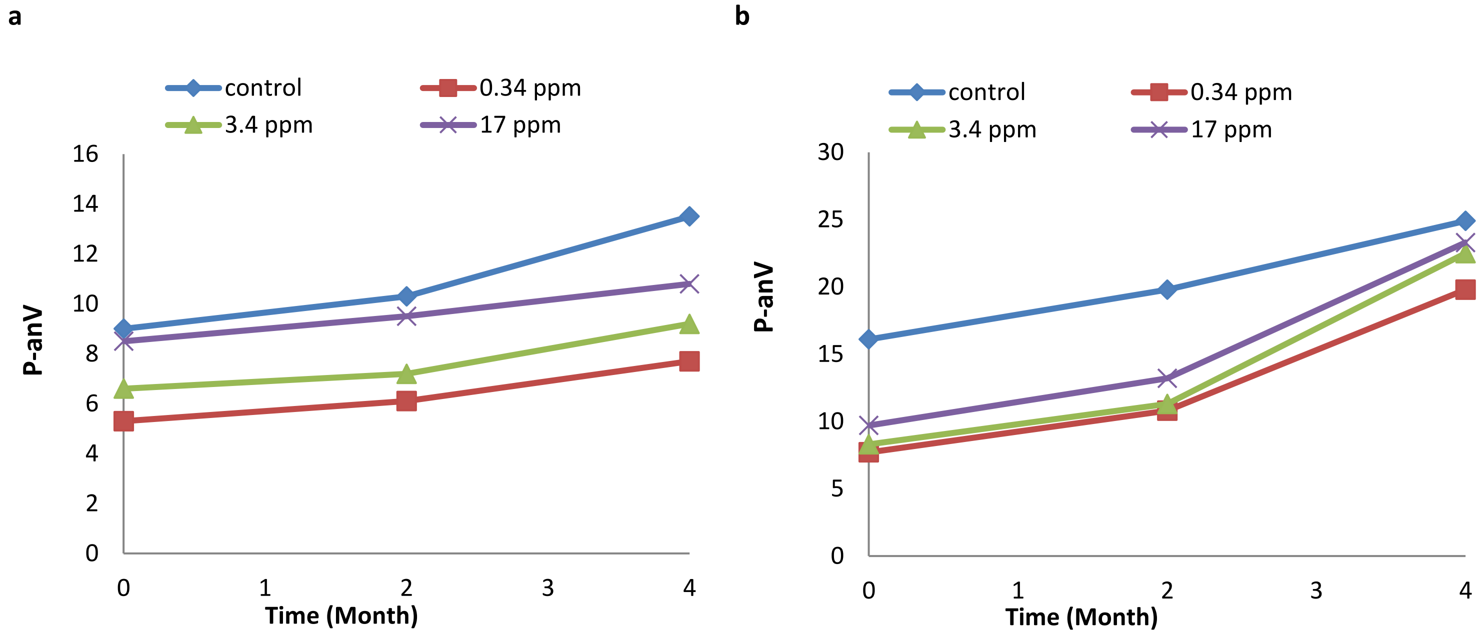

Figure (3): P-anV of Linseed oil (LO) with and without Ag NPs during storage in (a) dark, (b) Light

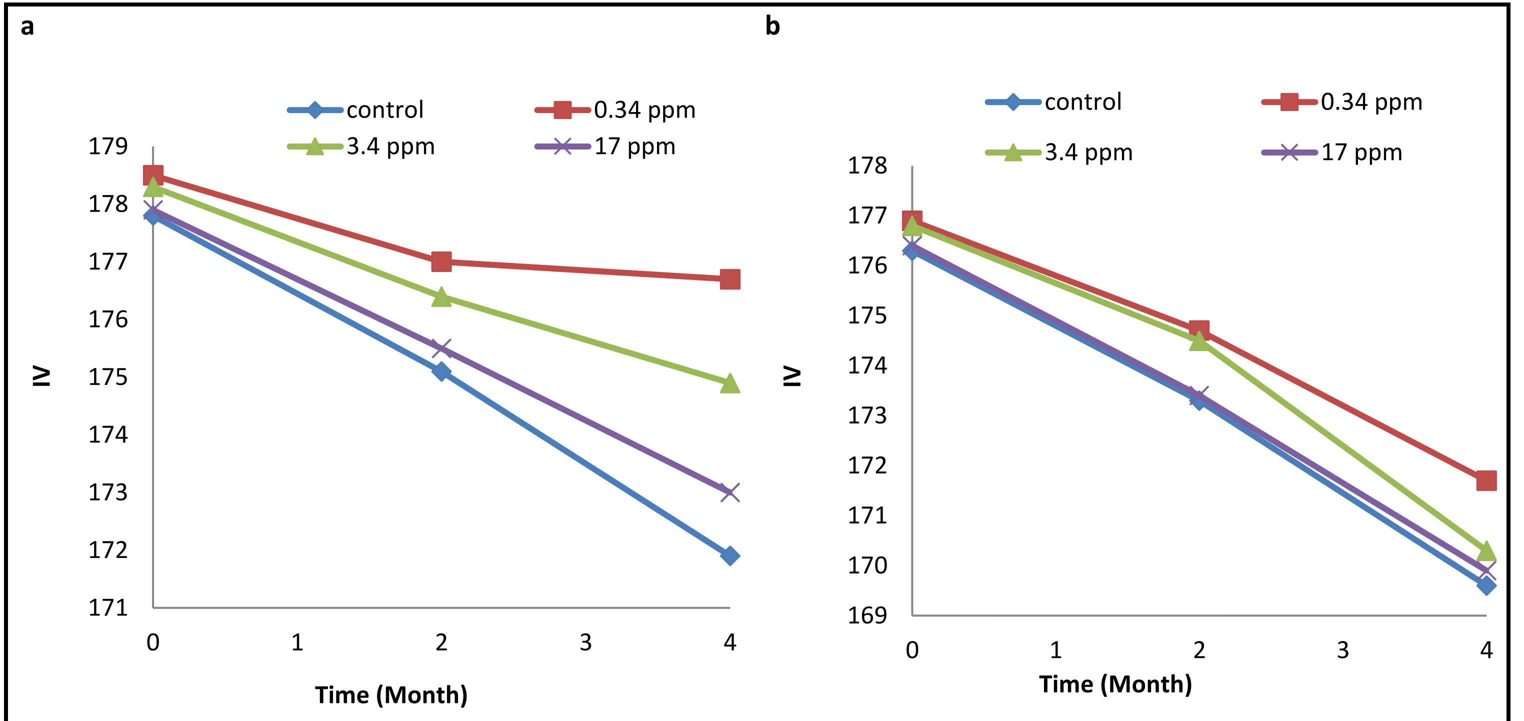

Figure (4): Iodine Value (IV) of Linseed oil (LO) with and without Ag NPs during storage in (a) dark,

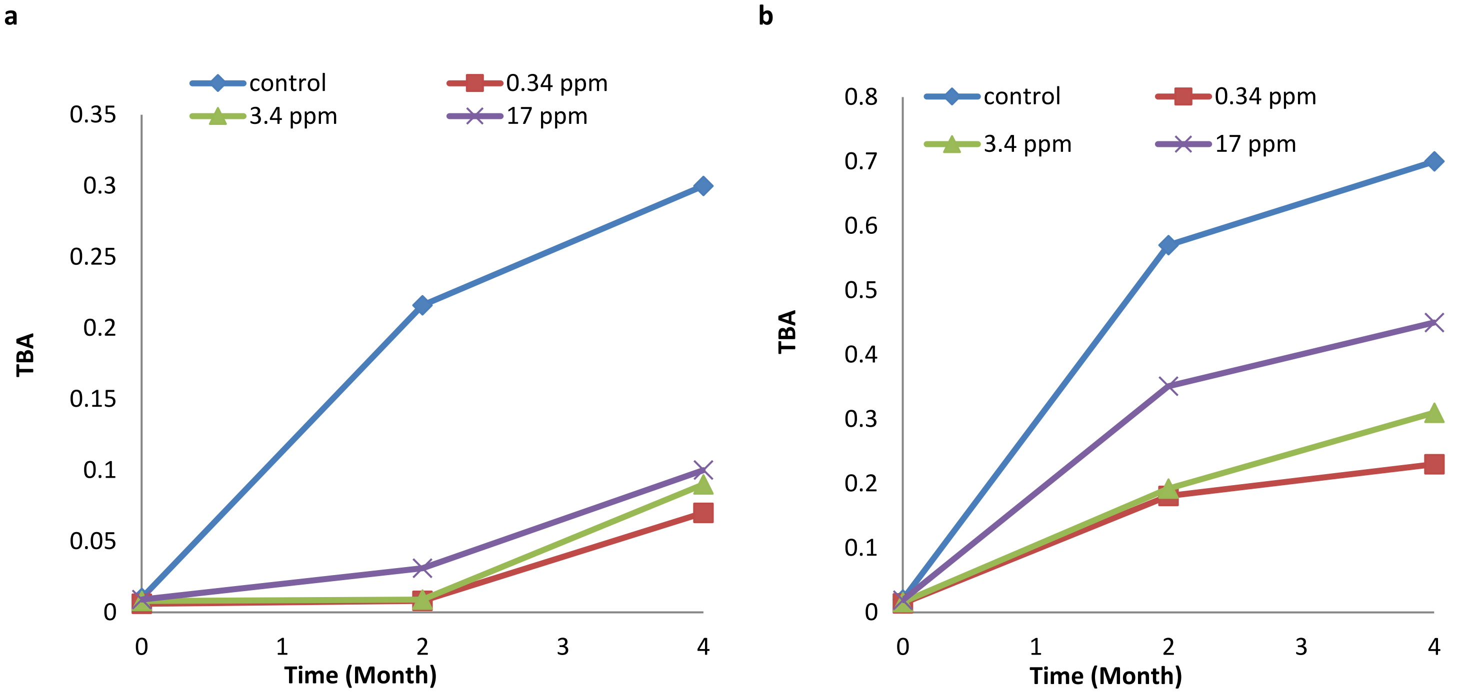

Figure (5): Thiobarbituric acid (TBA) value of Linseed oil (LO) with and without AgNPs during storage in (a) dark, (b) Light

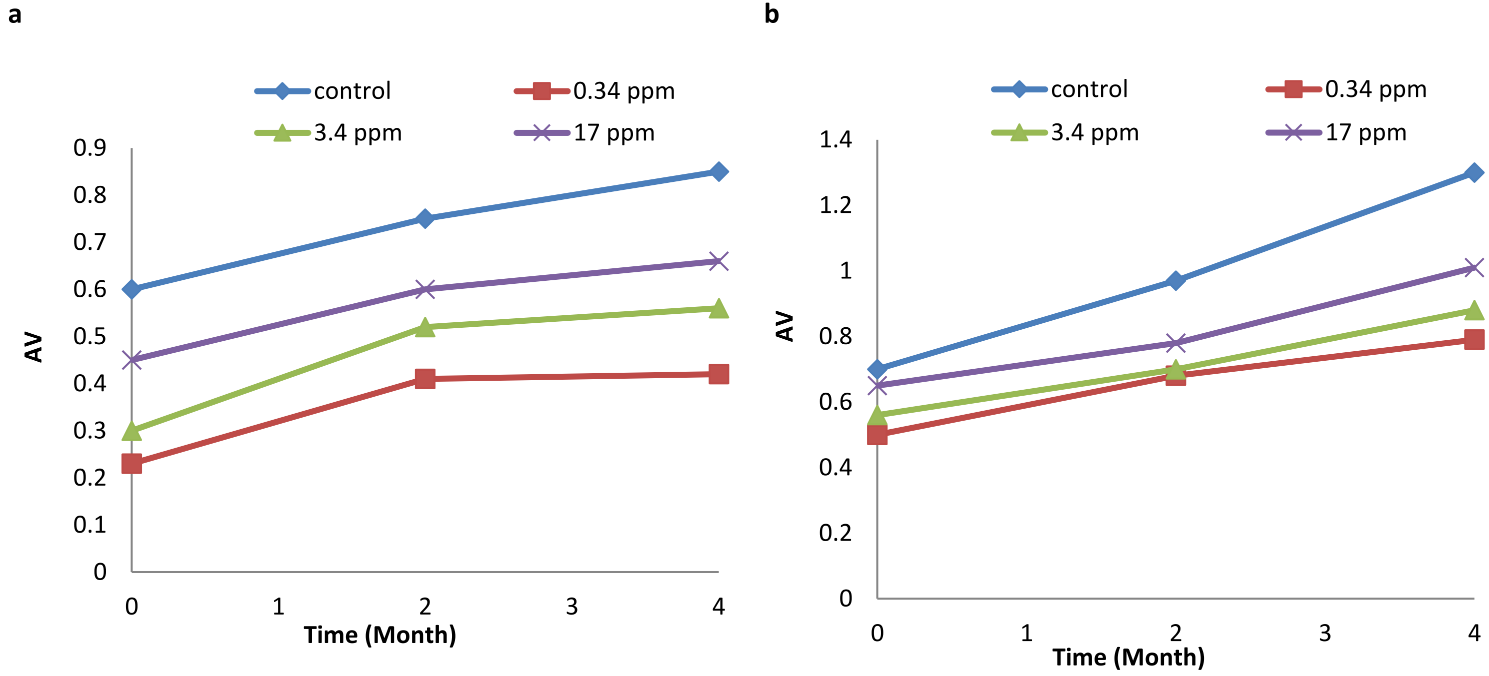

Figure (6): Acid Value (AV) value of Linseed oil (LO) with and without AgNPs during storage in (a) dark, (b) Light

Table 1: Microbiological analysis (total bacterial count) (no. of colonies × 10^2 CFU/mL) of LO subjected to dark and light storage with and without AgNPs through 4 months

Linseed Oil (LO)					
period	Condition of light	Control (0NP)	0.34 ppm	3.4ppm	17ppm
Starting time	Dark	0.2	-ve	-ve	-ve
	light	0.4	-ve	0.02	0.05
2 months	Dark	4.5	-ve	0.2	0.30
	light	10	-ve	5	10
4 months	Dark	11	-ve	0.3	0.5
	light	55	-ve	4	7

CFU, colony forming unit.

تحسين الخواص الفيزيائية والكيميائية لزيت بذر الكتان المعرض للضوء عن طريق المزج مع الجسيمات النانوية

اريج احمد ياسين [1], أميرة فوزى سليمان[2], نجلاء محمد عبد الفتاح [3]

[1] زميل تغذية وعلوم أطعمة, قسم تغذية اكلينيكة, المعهد القومى لأبحاث الأمراض المتوطنة و الكبد

[2] زميل كيمياء حيوية, معهد القلب القومى

[3] زميل تغذية, قسم الاحتياجات الغذائية والنمو, المعهد القومى للتغذية

الملخص العربى

من المثير للاهتمام معرفة أن جسيمات الفضة النانوية (Ag NPs) ليس فقط لها تأثير مضاد للبكتيريا، بل توفر أيضًا مستوى جيدًا من الأمان في مجالات أغذية الانسان. ولقد حصلت جسيمات الفضة النانوية على شهادة FDA في الولايات المتحدة وهي غير سامة وعديمة الرائحة وغير ضارة. توفر الاحتمالات المستقبلية لهذه المواد النانوية مجموعة متنوعة من الفرص لإنشاء سلع واستخدامات جديدة في النظام الغذائي. قد يكون لزيت بذر الكتان مدة صلاحية أطول إذا تمت إضافة جسيمات الفضة النانوية له كمواد حافظة بجرعات مختلفة. تم تحضير جسيمات الفضة النانونية بالطرق القياسية فى حمض الأوليك ثم قياس حجم وشكل جسيمات الفضة النانونية بواسطة الميكروسكوب الإلكتروني والأشعة فوق البنفسجية. خلال فترات التخزين، أجريت تقييمات للتزنخ والخصائص الميكروبيولوجية. أظهرت النتائج أنه في حين ترتفع مدة الصلاحية مقارنة بالعينة الضابطة، فإن جسيمات الفضة النانوية تقلل من العد الكلي للبكتيريا والتحليلات الكيميائية ما عدا قيم الرقم اليودى للعينات. يمكن أن تغير هذه التقنية تمامًا طريقة إنتاج الطعام. توفر آفاق هذه المواد النانوية العديد من الفرص لإنشاء سلع واستخدامات جديدة في السلسلة الغذائية.

الكلمات المفتاحية: جزيئات الفضة النانوية، زيت بذر الكتان، التزنخ.