

Green synthesis of ZnONPs using *Tradescantia spathacea* leaves extract and studying their thermodynamic effect on a mixture (2:8) (SDS/CTAB)

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Abstract

Abstract. In this study, zinc oxide nanoparticles were prepared using *Tradescantia spathacea* leaves extract; being an environmentally friendly and inexpensive material that has not been used previously for this purpose and characterizing it with both (XRD), (EDX) and (FESEM); and working on mixing (3 wt%) of the mixture Cetyltrimethylammonium bromide/Sodium Dodecyle Sulphate (CTAB/SDS) as a surfactant with ZnONPs as a tertiary mixture, with the aim of making CTAB and SDS more effective and with fewer damages by employment lower doses of them. effect of different concentrations of the zinc oxide nanoparticle solution on the ratio (2:8) was studied the most viscous of the binary mixtures of surfactants (SDS/CTAB), which is classified as containing more micelles than in other mixing ratios. The results showed that the highest value ($2705.223 \text{ Pa.s} \times 10^2$) was at a temperature of 298 K, and the lowest value ($1.506897 \text{ Pa.s} \times 10^2$) was at a temperature of 323 K, also showed that ΔG° values generally increased with increasing temperature and the higher concentration of nanomaterial gave lower ΔG° values. In addition, it was observed that the mixing exothermic process and the entropy change was negative, meaning that the system is in a state where the arrangement of molecules is regular.

Introduction:

The spread of nanotechnology has expanded to enter into several scientific fields, such as surface sciences, organic chemistry, and molecular biology [1], Semiconductor physics and energy storage [2,3] In addition to molecular engineering [4] and microelectronic manufacturing [5]. It may also include other applications such as advanced drug delivery systems [6] and vaccines [7] and incurable diseases such as cancer [8]. Green synthesis of nanoparticles using living cells via biological pathways is a more efficient technique and yields a higher mass compared to other related methods. Plants are sources of many biochemical

components and substances that can act as stabilizing and reducing agents for the synthesis of green nanoparticles. Green synthesis methods are environmentally friendly, non-toxic, cost-effective, and also more stable when compared to other biological, physical, and chemical methods [9].

The photocatalytic properties of nanozinc oxide facilitate the formation of reactive oxygen species (ROS) and thus allow used as additive active Antibacterial in dental [10], It also facilitates the isoelectric point of nanosized zinc oxide is the compatibility of surface protons with bodily fluids, a property that was designed in Drug carriers formulation [11].

"Properties of self - illumination" can be used in drug and photodynamic therapy for cancer[12], as well as operating surface of ZnO NPs prevents agglomeration, aggregation, and toxicity of Drug formulations in body Neighborhood and enhances targeted drug delivery[13,14], in addition to the surface, physical, chemical and properties of Zinc oxide nanoparticles directly affect on Physical absorption or Chemical absorption of solvent molecules and others in the interaction of chemical species or Biomolecules [15].

Zinc oxide surface atomic properties, arrangements and orientations are largely influenced by the type of synthesis methods used and the reaction conditions applied, which also affect the stability of the surface levels and toxicity [16,17].

Numerous researches have shown the possibility of green manufacturing of nano-zinc oxide; using orange fruit peel extract [18], Syzygium Cumini leaves extract [19] and Myrtus communis plant extract [20].

For active ingredients Surfaces wide use in drug delivery and Flotation agents [21]. Surface-active substances (CTAB/SDS) are substances that have the ability to reduce surface tension. Solutions of surfactant substances can be used helping to overcome biological barriers by forming micelles, which leads to increased randomness and decreased free energy of the system [22], thermodynamic study provides a scientific basis for understanding, improving, and designing the uses of surface tension reducers in various fields, by analyzing the equilibria and energies associated with the assembly and interaction of molecules [23].

Previous studies have shown that the surface action of ZnONPs with hydrophilic/hydrophobic materials improves dissolving the drug in the biological environment and enabling controlled release has also been shown to improve target penetration [24]. For example, scientists used Polyethylene Glycol, and Cyclodextrin as biomolecules to manufacture "hybrid materials" zinc oxide [25].

Many researches have revealed that nanofluids show stability and rapid ability to transfer heat and thermal conductivity [16,17]. They have shown promising solutions in Medicine [26], Food [20], Oil extraction [25,27], and treatment of Sewage [28], electronic applications [29], and solar thermal systems [30].

Surfactants improve Thermal Conductivity and Stability of Zinc oxide nanofluids [25]. By Adnan Qamar and others [31] been studied effects of (Sodium Hexametaphosphate) and acetyl acetone on aqueous Zinc oxide nanofluids as the results showed that nanofluids thermal conductivity based on sodium hexametaphosphate (SHMP) was above rate was

(23.70 %). Studied the stability of Zinc Oxide Nanofluids in the presence of (SDS) and Sodium dodecyl benzenesulfonate (SDBS) as reducing materials Muhammad and et al. [32]; They showed that zinc oxide nanofluids are stable at 95 °C with (SDBS). Jason et al [33] explained that nanofluids treated by Polymethyl Methacrylate (PMAA) have superior thermal conductivity and stability compared to pure nanofluids; which shows that adding a surfactant helps effectively improve the thermophysical properties of oxide liquids Nanoparticles of ZnO. Many studies have focused on "synergistic mixed surfactants" that are high surface activity and low surface energy [34,35,36], as the results showed that mixed surfactants lead to "higher reactivity and increased surface activity". Therefore, the current study aimed to add the active agent to the anionic/cationic interface CTAB/SDS (mixed) to reinforcement Metal Oxide Nanoparticle Dispersion, thermophysical features and stability for Nanofluids.

MATERIALS & METHODS

MATERIALS

SDS; formula (CH₃(CH₂)₁₀CH₂-OSO₃⁻Na⁺) (288.372 g/mol), CTAB; (C₁₉H₄₂BrN) (364.46 g/mol), sodium hydroxide, Aqueous zinc sulphate. This work also used both ethanol and *Tradescantia spathacea* plant.

MEASUREMENT OF VISCOSITY

The water bath is set to the temperature necessary for measurement, and the measurement is performed using a Pyknometer to measure density and viscometer for viscosity measurement equipped with an electronic stopwatch and a thermometer. The viscometer containing the model is immersed in the water bath for 5 minutes while stirring. The time of descent from a starting point to an end point is measured and the process is repeated 5 times. The average reading is calculated for each specific temperature:(293, 303, 313and323)K.

PREPARATION OF ZnONPs

Zinc oxide nanoparticles were prepared according to the following steps:

An aqueous extract of *Tradescantia spathacea* plant was prepared. On light heating (maintaining the temperature around 60 °C), a 1:1 mixture of the extract Plant is prepared and sodium hydroxide solution prepared by dissolving 8 g sodium hydroxide in 50 ml distilled water. Added to the previous mixture, at the same temperature, 50 ml of aqueous zinc sulphate solution 0.3 M, drops Just a drop with continuous stirring. It is observed that the color of the solution changes to a milky color with the formation of a precipitate.

The solution is filtered and the precipitate is washed with ethanol several times, then with deionized water, then dried in an oven drying at 60 °C for a day.

The work steps are as shown in the Figure (1).

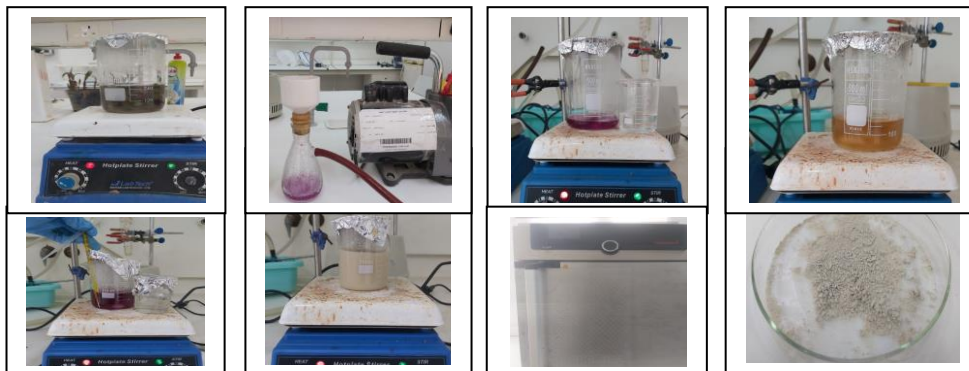


Fig 1. ZnONPs preparation steps.

PREPARATION OF SOLUTION

ZnONPs solutions

A concentration of (0.0001 M) was prepared as stock solution, by melting (0.0004 g) of nano-zinc oxide powder in distilled water, from which two other solutions were prepared with concentrations of (1×10^{-5} and 1×10^{-6} M).

Mixing solutions (ZnONPs/SDS/CTAB)

Number of solutions at certain concentrations were prepared as Tertiary blend consisting ZnONPs and SDS/CTAB. Preparation of (3 wt%) of SDS with solutions with the studied concentrations (1×10^{-4} , 1×10^{-5} and 1×10^{-6} M) of ZnONPs by dissolving (0.3093 g) in the concentration of the nanomaterial solution in volumetric vial (10 ml), and in the same way (3wt%) was prepared from CTAB, then the SDS/CTAB solution was mixed in the same concentration of the nanomaterial was at a ratio of (2:8), then the viscosity was tested and the thermodynamic functions were found at certain temperatures.

RESULT & DISCUSSION

Diagnosis of ZnONPs

X-Ray Diffraction

XRD analysis determines the long-range order of crystalline materials (structure) and the short-range order of non-crystalline materials. Most XRD analyzes focus on characterizing polymers, catalysts, and nanocomposites. Figure (2) shows the model under study, Using the English-origin XRD device.

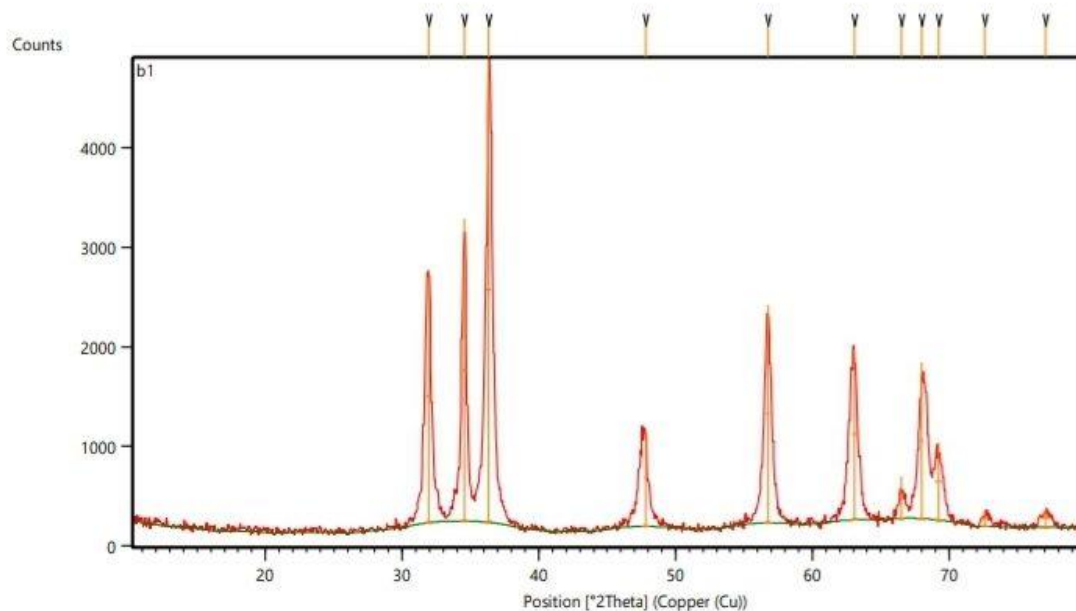


Fig 2. XRD measurement of the ZnONPs.

The results of XRD measurement shows that the crystalline size of the seven strongest bands of the nanomaterial is within the range (21.21 – 72.23) nm, which confirms that the size of the particles of the prepared material is within the nanoscale size. three largest values is (24.7, 24.6, 21.3) nm as shown in Table (1).

Tab 1. X-ray diffraction spectrum of zinc oxide nanoparticles.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width	Matched by
31.9572	2549.25	0.3936	2.80057	53.14	0.4723	
34.5789	3038.68	0.3444	2.59401	63.34	0.4133	
36.3494	4797.44	0.3444	2.47162	100.00	0.4133	
47.7992	977.71	0.1476	1.90291	20.38	0.1771	
56.7373	2189.27	0.2460	1.62254	45.63	0.2952	
63.0806	1713.56	0.3936	1.47379	35.72	0.4723	
66.5337	418.33	0.3936	1.40544	8.72	0.4723	
67.9986	1567.82	0.2952	1.37869	32.68	0.3542	
69.2307	780.13	0.5904	1.35713	16.26	0.7085	
72.6035	165.56	0.5904	1.30218	3.45	0.7085	
77.0453	187.48	0.7872	1.23781	3.91	0.9446	

Energy Dispersive X-ray (EDX)

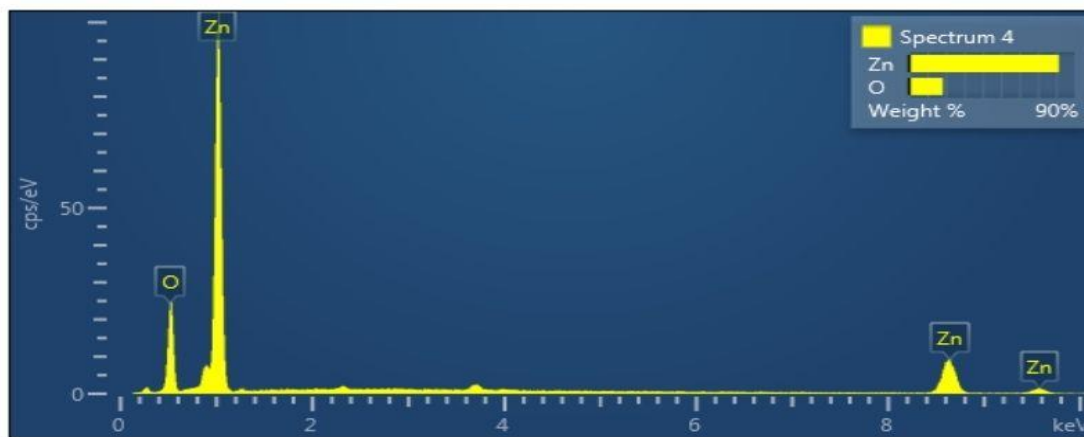
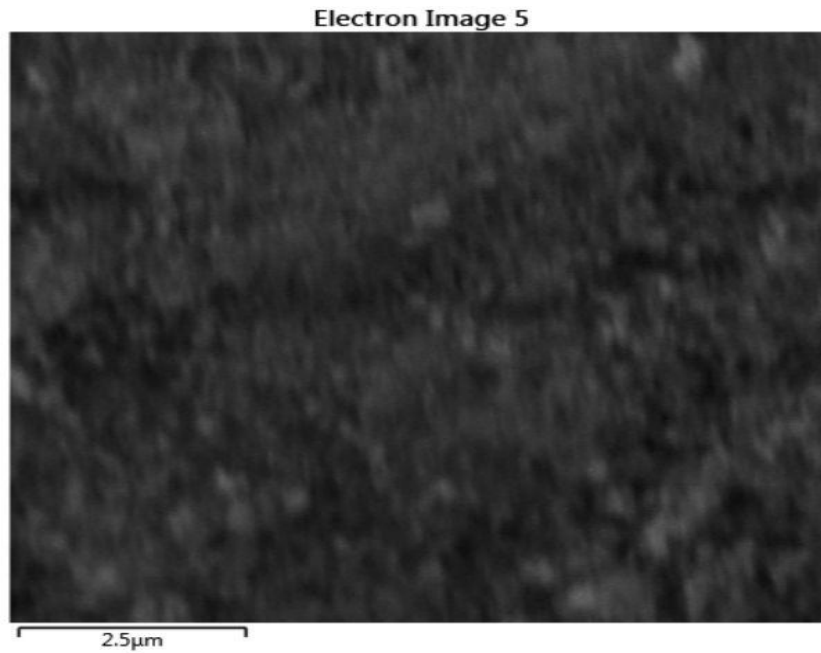


Fig 3. EDX measurement of the ZnONPs.

The X-ray energy dispersion diagram showed that the percentage of zinc represents 81.56 %, while the percentage of oxide represents 18.44 %.

Field-emission Scanning Electron Microscope (FESEM)

The resulting FESEM spectrum of the prepared ZnONPs shows that the material is in the form of small spherical aggregates, with an average nano-dimension of (19.31nm) at a magnification power of (100 nm).

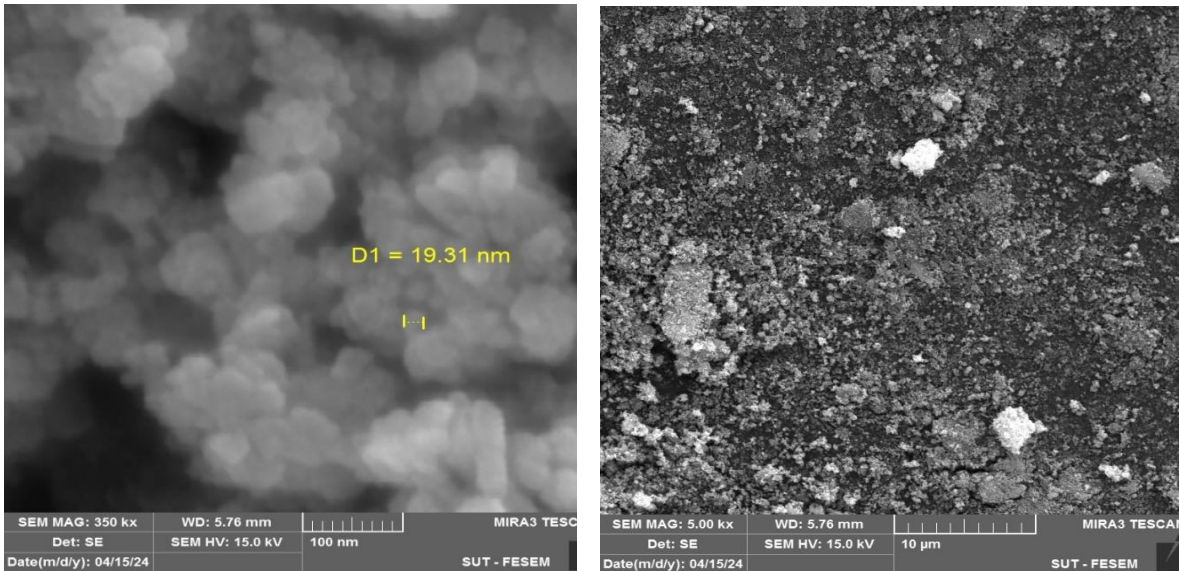


Fig 4. FESEM spectrum of the ZnONPs.

Viscosity and Thermodynamic functions of mixture (ZnONPs/SDS/CTAB)

Aqueous solutions of surfactants have distinctive flow and flow characteristics due to their high viscosity characteristics as a result of the presence of molecular interaction forces, the most important of which are the forces of attraction between the tail groups, the hydrophobic effect, in addition to the forces of electrostatic attraction between the head groups with opposite charges. As a result, the presence of additives in such systems plays a role. It is important in two opposite ways: it either maintains or enhances the flowable and flowable properties of these systems, or it disintegrates the worm-like micelles, leading to a reduction in their viscosity and a decrease in their flowable properties. In both cases, this depends on the nature of the additive and its concentration [37].

Complex formation as Micelles or Monomers between Nanoparticles and Surfactants can significantly improve the stability of dispersion systems, as Emulsions and Foams have proven successful in practice by many researchers [38,39,40,41].

NPs presence reduces surface tensions and accelerates Surfactant Adsorption. Surfactants can adsorb at interfaces and change them, thus "the surface of nanoparticles is more active compared to regular surfaces". This has been proven through numerous scientific studies [42,43].

Increasing the concentration of nanofluids and decreasing the temperature lead to an increase in viscosity. Because at high temperatures, particles can overcome the forces that hold them because there is thermal energy, enhancing fluid movement and reducing η [44]. This makes the use nanofluids possible at high temperatures, thus taking advantage of lower η . As shown in the current study, Table (2).

Tab 2. Viscosity of blend (ZnONPs/SDS/CTAB) at certain concentrations and different temperatures.

Con. ZnONPs With 3wt% (SDS/CTAB) (2:8)	$\eta \times 10^2$ (Pa.s)			
	T=293 K	T=303 K	T=313 K	T=323 K
1×10^{-6} M	2247.554	21.39388	5.346193	1.506897
1×10^{-5} M	2350.227	24.34958	8.493637	1.803873
1×10^{-4} M	2705.223	27.48495	10.73078	2.27055

It turned out that the higher concentration gave better results and higher η than the rest of the measured concentrations, as shown in the figure (5).

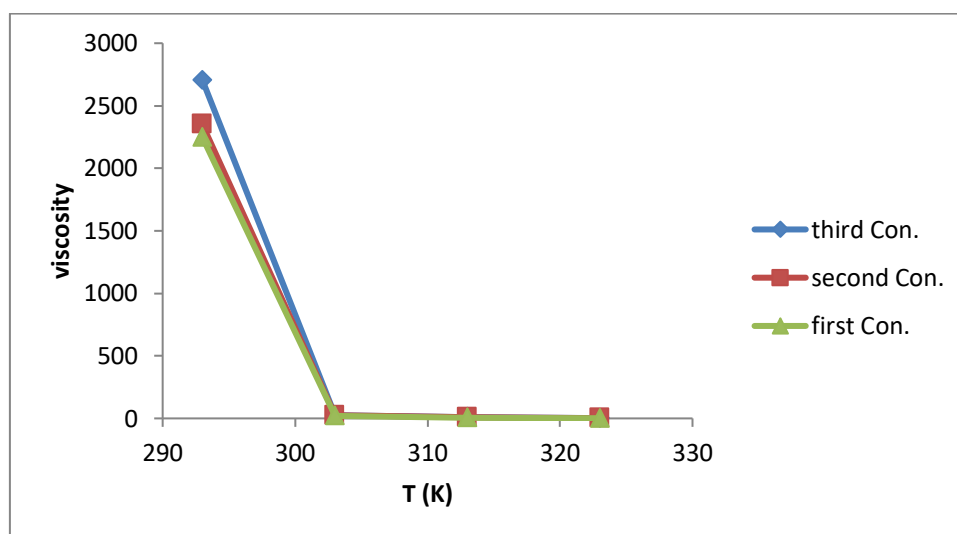


Fig 5. Relationship viscosity of triple blend with temperature at different concentrations of (ZnONPs).

The following equations were used to obtain the thermodynamic functions:

$$\Delta G^{\circ} \approx -RT \ln(\eta/2 \times 10^{-3})$$

$$\frac{d \ln(\eta/2 \times 10^{-3})}{d(1/T)} \approx \frac{-\Delta H^{\circ}}{R}$$

$$\Delta S^{\circ} = \frac{\Delta H^{\circ} - \Delta G^{\circ}}{T}$$

Studies by Nguyen et al. [45] and Pastoriza Gallego et al. [46] have proven this. Nguyen et al [45] also reported that the dynamic η increases with nanoparticle size. ΔG° of the blend was also studied, which gave in Table No (3).

Tab 3. ΔG° for blend (ZnONPs/SDS/CTAB) at different temperatures.

Con. ZnONPs With 3wt% (SDS/CTAB) (2:8)	ΔG° (kJ/mol)			
	T=293 K	T=303 K	T=313 K	T=323 K
1×10^{-6} M	-22.7207	-11.7708	-8.55064	-5.42318
1×10^{-5} M	-22.8295	-12.0968	-9.75532	-5.90625
1×10^{-4} M	-23.1722	-12.4019	-10.3637	-6.52413

It was observed that increasing temperature leads to increasing ΔG° values for the mixture of surfactants, this is because increasing the temperature increases the free energy in the SDS/CTAB/ZnONPs system because it causes an imbalance between the regulating forces (electrostatics, surface action, particle packing), which raises the enthalpy of the system more than the entropy gain, thus increasing ΔG° [47]. the higher concentration of the nanomaterial gave lower free energy values, as the ΔG° value was (-23.1722 KiloJoules/mole) at 293K and (1×10^{-4} M) while giving the highest value (-5.42318 KiloJoules/mole) at 323K and (1×10^{-6} M).

By drawing a graphic relationship between ($1/T$) and ($\ln \eta$), the values of enthalpy ΔH° can be calculated, the slope, intercept, and R^2 (Correlation Coefficient) can be found. According to the results, triple blend process (ZnONPs/SDS/CTAB) is exothermic process at all concentrations of the nanomaterial prepared with the mixture, and its value increases by increasing focus, where ΔH° is (-176.0573 KiloJoules/mole) at (1×10^{-4} M), as shown in Table (4).

Tab 4. ΔH° of blend of SDS/CTAB (3wt%) with ZnONPs in different concentrations.

Con. ZnONPs With 3wt% (SDS/CTAB) (2:8)	ΔH° (kJ/mol)	Slope	Intercept	R^2
1×10^{-6} M	-176.0573	21176	-63.631	0.8998
1×10^{-5} M	-179.0420	21535	-64.980	0.9070
1×10^{-4} M	-185.0364	22256	-67.528	0.9061

Since the mixing process produced heat, the entropy change is negative, meaning that the system is in a state where the arrangement of molecules is regular. As shown in Table (5). Which can be represented graphically as in Figure (6).

Tab 5. ΔS° of (SDS/CTAB) blend in presence ZnONPs at different T.

Con. ZnONPs With 3wt% (SDS/CTAB) (2:8)	ΔS° (J/mol.K)			
	T=293 K	T=303 K	T=313 K	T=323 K
1×10^{-6} M	-107.491	-146.189	-157.718	-168.246
1×10^{-5} M	-101.126	-139.119	-147.875	-160.756
1×10^{-4} M	-96.9714	-135.127	-142.946	-155.859

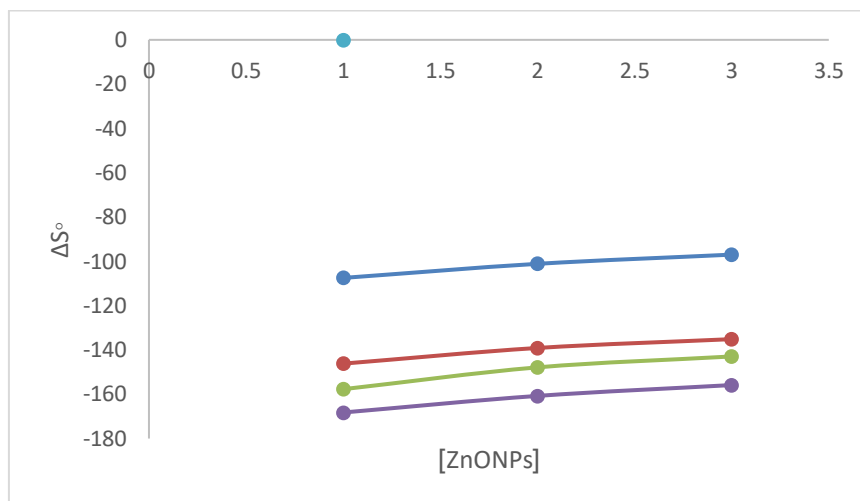


Fig 6. Relationship ΔS^0 of triple blend with concentrations of (ZnONPs) at different temperature.

The higher concentration of the nanomaterial gave higher Random values, the ΔS^0 value was (-96.9714 KiloJoules/mole. Kelvin) at 293K and (1×10^{-4} M) while giving the lower value (-168.246 KiloJoules/mole. Kelvin) at 323K and (1×10^{-6} M); As a result of the large interaction force between molecules.

CONCLUSION

- Increasing temperature leads to disintegration of molecular interaction forces and therefore increase kinetic energy of the molecules and decrease in viscosity.
- Presence of micelles is a positive factor in improving the effectiveness of the zinc oxide nano solution, the reason is to increase the stability of nanofluids.
- Increasing nanomaterial concentration led to decrease in ΔG^0 in the ternary blend (CTAB/SDS) with (ZnONPs); this is due to quantum physics and the unique properties of nanomaterial.
- The process is exothermic at the lower nanosolution concentration.
- The values of ΔS^0 for the ternary mixture increase with increasing concentration of nano-zinc oxide at different temperatures, which means increase in physical interactions and composition micelles; Which means that the mixture of materials has become more regular

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التخليق الأخضر لـ ZnONPs باستخدام مستخلص أوراق نبات *Tradescantia spathacea* ودراسة تأثيرها الديناميكي الحراري على مزيج (SDS/CTAB) (2:8)

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الخلاصة:

تم في هذه الدراسة تحضير دقائق اوكسيد الزنك النانوية باستخدام نبات *Tradescantia spathacea* و تشخيصها بكل من حيود الاشعة السينية (XRD) و تشتت الطاقة بالاشعة السينية (EDX) والعمل على مزج (3wt%) من مزيج (SDS/CTAB) كمواد فعالة سطحياً مع الـ ZnONPs, و الذي يعتبر احد المواد النانوية المهمة المستخدمة في تطبيقات صناعية عديدة كصناعة الخلايا الشمسية, في معالجة المياه, المبيدات الحشرية, فضلاً عن اثره المهم من الناحية الطبية كتطبيقه في صناعة العظام, مضاداً للسرطان و في توصيل الادوية, إضافة لإستخدامه في قتل الكائنات الحية الدقيقة الضارة في العبوات, وفي المواد الواقية من الأشعة فوق البنفسجية مثل المنسوجات كمزيج ثلاثي. بهدف جعل كل من SDS, CTAB ذات فعالية اعلى و اثار جانبية اقل و ذلك عن طريق استخدام جرعات اقل منها, إذ تم دراسة تأثير تراكيز مختلفة لمحلول مادة اوكسيد الزنك النانوية على النسبة (2:8) الأكثر لزوجة للمزيج الثنائي للمواد الفعالة سطحياً (SDS/CTAB) و الذي يُصنّف على انه يحوي كمية من المذيلات اكثر مما هو عليه في نسب المزج الأخرى. وبيّنت النتائج ان القيمة الأعلى كانت ($2705.223 \text{ Pa.s} \times 10^2$) عند درجة حرارة 298 K والقيمة الأدنى ($1.506897 \text{ Pa.s} \times 10^2$) عند درجة حرارة 323 K. كما بيّنت النتائج زيادة قيم ΔG° عموماً مع زيادة درجة الحرارة وان التركيز الأعلى للمادة النانوية اعطى قيم ΔG° اقل. إضافة لذلك لوحظ ان عملية المزج كانت باعثة للحرارة وأن تغيّر الإنتروبي يكون سالب, اي ان النظام يتحوّل الى حالة يكون فيها ترتيب الجزيئات اكثر إنتظاماً.

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مواد فعالة سطحياً، سوانل نانوية، ZnONPs،

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