

Stability of alkyd emulsion resins and anti-corrosion properties of water-based alkyd emulsion paints

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Abstract:

Vegetable oils extracted from nuts are a useful source of raw materials for the paint industry. Alkyd resins were synthesised with a content of 50 wt% soybean oil. The synthesised alkyd resins were evaluated by Fourier transform infrared spectroscopy (FTIR) and gel permeation chromatography (GPC). Particle size was determined using dynamic light scattering (DLS). Soybean oil alkyd resins were emulsified with the anionic surfactants linear alkylbenzene sulfonate (LAS)/sodium lauryl sulphate (SLS) and the nonionic surfactant nonyl phenol ethoxylate (NP9) in an aqueous solvent. The anti-corrosion properties of the alkyd emulsion coatings were determined using the electrochemical method. The results showed that with a content of 13 wt% of the mixture of LAS and NP9 surfactants, the formed alkyd emulsion resins had good stability. Alkyd emulsion paint with a water solvent achieved the physicochemical properties required for steel protection. However, the anti-corrosion properties of the fabricated alkyd emulsion coatings need to be improved. The use of water as a solvent is gradually replacing organic solvents, with many prospects for new applications.

Keywords: alkyd emulsion resin, emulsifiers, soybean oil alkyd resin, surfactants water-based alkyd emulsion paint.

Classification numbers: 2.1, 2.2, 2.3

1. Introduction

The paint industry is developing in an environmentally friendly direction. Vegetable oils extracted from nuts are a useful source of raw materials for the paint industry [1, 2]. Polyester resins modified with vegetable oils form alkyd resins. Thus, alkyd resins are easier to biodegrade and more environmentally friendly than other synthetic polymers [3, 4]. Drying oils and semi-drying oils such as sunflower oil, castor oil, soybean oil, and linseed oil are often used to modify polyester resins for paint [5, 6].

Solvent-based alkyd resins have a high content of volatile organic compounds (VOCs). These compounds pollute the environment. Consequently, paint manufacturers have reduced the VOC content in paints [7, 8].

In one study, the authors prepared water-based acrylic alkyd resins. These resin systems are stable, with a dispersion of 32 compared to a conventional resin of 8 [9]. Many factors affecting the stability of water-based acrylic alkyd resins were examined [10].

The properties of surfactants in water-based acrylic alkyd emulsion resins were also studied [11].

In another study, the authors synthesised alkyd resins from soybean oil and glycerol using a Zirconium Octoate catalyst [12].

In other studies, the authors have also studied the use of aqueous solvents as substitutes for organic solvents in paints [13-15].

The stability of alkyd emulsion resin depends on the emulsifier and emulsification conditions. Water-based alkyd emulsion paints are environmentally friendly. However, previous results showed that the stability of the emulsion system needs to be improved, and efforts must continue to enhance the durability and corrosion resistance of emulsion alkyd paints.

This study presents the effects of the NP9 nonionic emulsifier and the LAS/SLS anionic emulsifier on the stability of alkyd emulsion resins and water-based alkyd emulsion paint.

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2. Materials and methods

2.1. Materials

Glycerol is a liquid with a density at 25°C of 1.261 g/cm³; its boiling point is 290°C; its purity is 99.5% (Duc Giang Chemical, Vietnam). Phthalic anhydride is a white crystal with a density at 25°C of 1.53 g/cm³; a melting point is 131.6°C, a boiling point is 295°C, purity is 99.5% (Shandong Hongxin Chemical, China). Soybean oil is liquid, iodine index (Wijs) is 124-139 (Tuong An, Vietnam). Lead oxide (PbO) is a red crystal with a density of 9.53 g/cm³, a melting point is 888°C, purity is 98% (Jiangsu Tianpeng, China). Xylene (99.5% purity, China), methanol (99.5% purity, China), acetone (99.5% purity, China), and phenolphthalein (99.5% purity, China).

Emulsifiers include NP9 nonionic surfactant. NP9 is a liquid with a density of 1.055 g/cm³ (at 20°C), boiling point is 293-297°C, purity is under 99% (Tergitol, Dow, Indonesia). Anionic surfactant is SLS. SLS is a white solid, melting point is 206°C (99% purity, Disponil SLS 103, China). Anionic surfactant is LAS, density is 1.06 g/cm³, boiling point is 315°C. LAS is a thick liquid with a dark brown colour, soluble in water (99.8% purity, Henan Surface Chemical Industry, China).

Dispersing additive is CFC-690H, (Cheongwoo CFC, Korea). Titanium dioxide powder (Tiona 595, China), talcum powder (Talc 1250, Taiwan), calcium carbonate filler (China). Defoamer is BYK024 (Germany). Octa-Soligen 144 aqua drying agent is a purple viscous liquid with metal content in the mixture: Co²⁺: 0.95-1.05%, Zn²⁺: 3.10-3.30%, Zr²⁺: 7.00-7.40% (Borchers GmbH, Germany). Acrysol thickener RM-825 (Dow, USA).

2.2. Preparation of samples

Alkyd resin synthesis:

Alkyd resin (A0) reaction consists of three stages [16, 17]:

1. Oil triglycerides will react with glycerol at 220-240°C with Pb²⁺ (PbO) catalyst to form monoglycerides.
2. Monoglycerides react with phthalic anhydride at 170°C to form monoester.
3. The monoester condensation reaction continues at a temperature of 220-240°C to form alkyd resins.

Using xylene solvent attracts water to increase the performance of the reaction. The ratio of xylene/water is 4/1 (wt/wt). The stopping time of the reaction is when the acid index (AI) of the mixture is less than 30.

The alkyd resin reaction is presented in Fig. 1.

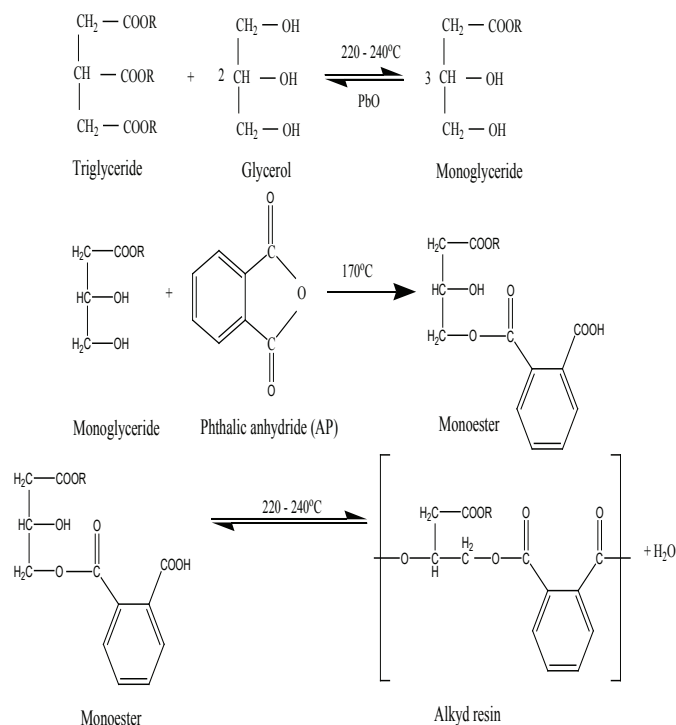


Fig 1. Alkyd resin reaction from glycerol and phthalic anhydride (AP).

Soybean oil (50.00 g) and glycerol (16.02 g) were placed in a 500-ml glass reaction tank. The reaction was heated to 170°C and afterwards, 0.2 g of PbO catalyst was added. The temperature of the reaction was raised to 220°C to continue with the reaction of converting triglycerides into monoglycerides. After a reaction time of 1 h, the solubility of the mixture in ethanol prepared according to an ethanol/mixture ratio of 3/1 (v/v). The reaction temperature was reduced to 170°C and phthalic anhydride (AP) (38.67g) was added to the reaction mixture to conduct a monoester reaction for a period of 1.5 h. Then, the reaction was heated to 220°C to continue the condensation reaction. The xylene solvent (18.81 g) was slowly added to the mixture to separate the water. The reaction ended when the acid index was less than 30 mg KOH/g.

Synthetic method of alkyd emulsion resin:

Emulsification method (phase reversal method): The emulsifiers used are a nonionic mixture of NP9 and the anionic LAS/SLS.

Alkyd resin is placed in a 250 ml glass beaker and heated to 70°C. LAS/SLS and NP9 emulsifiers are added, and the mixture is stirred at 500 rpm for 10 minutes. Water is added slowly at a ratio of 50:50 (v/v) to the emulsifier. The mixture temperature is increased to 90°C, and the stirring speed is increased to 2000 rpm for 45 minutes. Then, the mixture temperature is reduced by 30°C to stabilise and store.

Effect of surfactant content on the stability of emulsion alkyd resins: The surfactant content affects the emulsification ability and properties of the emulsion system. The suitable surfactant content ranges from 10 to 16 wt% [9-12]. After the emulsification process was completed, the samples were left to stabilise at room temperature for 24 h and evaluated for the stability of the alkyd emulsion resin (Table 1).

Table 1. Effect of surfactant content on stability of alkyd emulsion resin.

Specifications	Samples							
	S1	S2	S3	S4	S5	S6	S7	
Surfactant content (wt%)	10	11	12	13	14	15	16	
Ingredients	Alkyd resins (g)	40.00	39.00	38.00	37.00	36.00	35.00	34.00
	SLS anionic surfactant (g)	3.33	3.67	4	4.33	4.67	5.00	5.33
	NP9 nonionic surfactant (g)	6.67	7.33	8	8.67	9.33	10.00	10.67
	Water (g)	50.00	50.00	50.00	50.00	50.00	50.00	50.00
Parameters for evaluating the stability of the emulsion	- Ability to create an emulsion system - Solid content - pH - Room temperature storage							

Effect of surfactant type on the stability of alkyd emulsion resin: Anionic surfactants provide the ability to emulsify but do not support the storage process of the product. Nonionic surfactants, however, participate in both the emulsification process and the storage. Therefore, choosing the correct surfactant ratio is essential for both the emulsification process and the properties of the emulsion system. The results of the study showed that if the surfactant content was 13 wt%, the alkyd emulsion resin had the best stability (Tables 1 and 5).

The content of the emulsifier is 13 wt%. The ratio of emulsifiers SLS:NP9 is 1:2 (wt/wt) (AE1 emulsion alkyd resin sample) and LAS:NP9 is 1:2 (wt/wt) (AE2 emulsion alkyd resin sample) with the following preparation menu (Table 2).

Table 2. Samples of alkyd emulsion resin.

Ingredients	Weight (g)	
	Alkyd emulsion resin (AE1)	Alkyd emulsion resin (AE2)
Alkyd resin (A0)	37.00	37.00
SLS anionic surfactant	4.33	-
LAS anionic surfactant	-	4.33
NP9 nonionic surfactant	8.67	8.67
Water	50.00	50.00

Water-based alkyd emulsion paints:

Water-based alkyd emulsion paints are presented in Table 3.

Table 3. Water-based alkyd emulsion paints.

Ingredients	White alkyd emulsion paint weight (g)
Water	32.56
CFC-690H dispersing additive	0.72
Tiona 595 titanium dioxide powder	45.66
CaCO ₃ filler	13.50
Talc powder	7.02
BYK024 deformer	0.54
Total of base part	100
Alkyd emulsion resin (AE1)	68.87
Base part	26.67
Soligen 144 Aqua drying agent	1.85
BYK024 deformer	0.75
RM825 thickener	0.75
Water	1.11
Total	100

The composition of water-based alkyd emulsion paint includes alkyd emulsion resins, emulsifier systems, aqueous solvents, pigments, fillers, additives, and drying agents. These ingredients were crushed in a ball machine for 4 h to reach the fineness required for the paint.

2.3. Analytical devices and methods for the analysis

The acid index of the resin is determined according to ASTM D1639-90. The solid content of the resin is measured according to ASTM D2369-98. The molecular mass of the resin is determined using GPC gel chromatography (Cirrus GPC version 3.0). The physicochemical properties of the resin and the mechanical properties of polymer films are assessed according to ASTM and TCVN standards. Functional groups in alkyd resins are identified using FTIR infrared spectroscopy. The stability of the emulsion system is evaluated according to the warm temperature storage method and room temperature according to the JIS K 5400 standard. Particle size and particle size distribution in suspensions are measured by dynamic light scattering (DLS). The anti-corrosion properties of paint films are evaluated electrochemically according to ASTM G5-94, G102-189, and G106-89 (Biologic model VMP3B-5).

3. Results and discussion

3.1. Synthesis of alkyd soybean oil resin

Determination of the acid index of soybean oil alkyd resins:

Alkyd resins are synthesised with a content of 50 wt% soybean oil (sample A0). The results of determining the acid index (AI) of the alkyd resin are presented in Fig. 2.

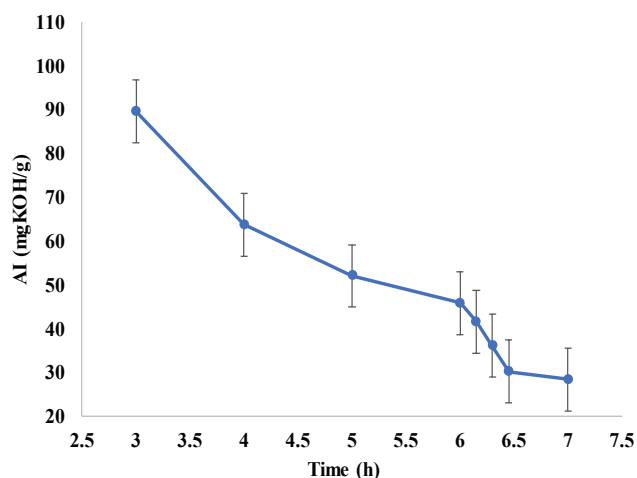


Fig 2. Acid index of soybean oil alkyd resin (sample A0).

From Fig. 2, the acid index rapidly decreased during the reaction phase from 89.6 to 52.1 mg KOH/g after 2 h. After 7 h of reaction, the acid index dropped from 45.9 to 28.46 mg KOH/g. The acid index is the number of mg of KOH used to neutralise all the acid in the sample. A low acid index indicates a low amount of residual acid in the resin, showing that the alkyd resin reaction has occurred with high efficiency and all the acid has reacted. The alkyd resin reaction follows the mechanism of polymerisation, where functional groups react with each other, and the reaction occurs over a long period [1-4].

The results of FTIR analysis of soybean oil-modified alkyd resins (A0 sample) are presented in Fig. 3.

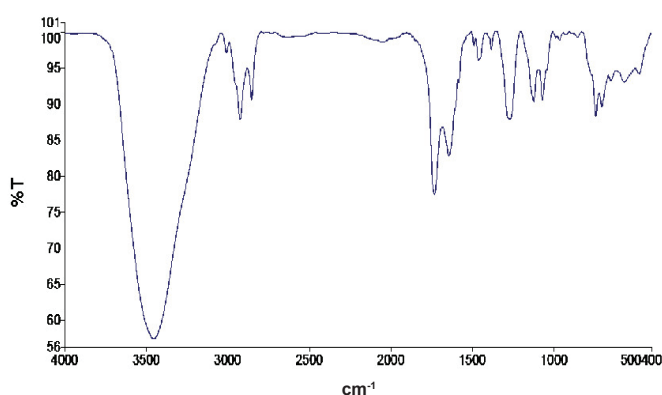


Fig 3. Fourier transform infrared spectrum of soybean oil alkyd resin (sample A0).

The results of infrared analysis from Fig. 3 show that alkyd resins have characteristic peaks. The strong absorption valence oscillation peak at 3455.36 cm⁻¹ is characteristic of the free hydroxyl group (-OH group) in the glycerol molecule of unsaturated fatty acids in soybean oil. The absorption peak at 3010.45 cm⁻¹ is characteristic of the double bond of the -C=C- group without conjugation of linoleic acid in soybean oil. The oscillation peak at 1732.85 cm⁻¹ with strong absorption intensity is characteristic of the C=O group. The oscillation peak at 1122.61 cm⁻¹ is characteristic of the -CO group attached to the phenyl radical. The oscillation peak at 1582.56 cm⁻¹ is characteristic of the -C=C- group in the aromatic ring of phthalic anhydride. The absorption peak at 1270 cm⁻¹ characterises the ester group (-COO-).

Thus, from FTIR spectroscopy, it is shown that the soybean oil alkyd resin (A0 sample) has fully demonstrated characteristic functional groups.

The physical chemistry properties of synthetic soybean oil alkyd resins are presented in Table 4.

Table 4. Properties of soybean oil alkyd resin.

Specifications	Alkyd resin (sample A0)
Solid content (%)	98.2
Viscosity (25°C, seconds)	83
Acid index (mgKOH/g)	28.46
Molecular mass (g/mol)	M _n 1.9144e ³
	M _w 6.2033e ³
Polydispersity (PDI=M _w /M _n)	PDI=3.24

The results from Table 4 showed that the soybean oil alkyd resin (A0 sample) had a low acid index (28.46 mgKOH/g), showing a high efficiency of the reaction and that the reaction occurred completely. If the acid index of alkyd resins is less than 30, then they can be applied to make paint [1, 5, 10]. Alkyd resin A0 has an average molecular mass of M_n=1.9144.e³ g/mol, a molecular weight of M_w=6.2033.e³ g/mol, and a polydispersion index of (PDI=M_w/M_n)=3.24. According to another study [10], the polydispersion of alkyd resins appropriate for making paint is under 8. The polydispersion index indicates the uniform molecular mass level of polymers. Soybean oil alkyd resin A0 is suitable for paint application [2, 5, 7, 9, 10].

3.2. Properties of alkyd emulsion resins

Effect of surfactant content on the stability of alkyd emulsion resins:

The effect of surfactant content on the properties of the emulsion system is presented in Table 5.

Table 5. Effect of surfactant content on the stability of alkyd emulsion resins.

Sample	Surfactant content, wt%	Parameter		
		Solid content, wt%	Pre-storage	Post-storage
S1	10	53.1	pH=3	pH=2 The emulsion system is unstable. The system is separated into resin and water after 1 day
S2	11	51.7	pH=4	pH=3 The emulsion system is unstable. The system is separated into 2 layers: water and emulsion, homogeneous when stirred. The system is separated into resin and water after 1 week
S3	12	52.8	pH=4	pH=4 The emulsion system is unstable. The system is separated into 2 layers: water and emulsion, homogeneous when stirred. The system is separated into resin and water after 2 weeks
S4	13	52.6	pH=4	pH=4 The emulsion system remained stable after 3 weeks
S5	14	49.7	pH=4	The emulsion system is unstable. The system is separated into 2 layers: water and emulsion, homogeneous when stirred. The system is separated into resin and water after 3 weeks
S6	15	48.3	pH=4	pH=4 The emulsion system is unstable. The system is separated into 2 layers: water and emulsion, homogeneous when stirred. The system is separated into resin and water after 2 weeks
S7	16	-	pH=5	pH=3 The emulsion system is unstable. The system is separated into resin and water after 3 days

The results from Table 5 show that all samples were capable of producing alkyd emulsion resins. When stirring ceased, the samples were foamy, the mixture was milky white, and the emulsion alkyd resin could form films. Upon starting storage at room temperature, the samples began to show instability; the system separated the resin from the mixture. Samples S1, S2, and S3 had an unstable emulsion system, with resin separation appearing after 1 day to 2 weeks. Samples S5, S6, and S7 had a storage time of more than 2 weeks but still showed instability. Sample S4 had a storage time of about 3 weeks, and the system remained stable with no separation phenomenon observed.

Therefore, the S4 alkyd emulsion resin sample demonstrated the best stability with a suitable surfactant content of 13 wt%.

The stability of the emulsion alkyd resin based on emulsifiers:

The stability of emulsion alkyd resins based on emulsifiers is presented in Table 6.

Table 6. The stability of alkyd emulsion resins.

Specifications	Alkyd emulsion resin AE1	Alkyd emulsion resin AE2
Solid content	52.6 wt %	48.35 wt%
Stage	The mixture is milky white	The mixture is milky white
Pre-store at room temperature	pH=4	pH=4
After storage at room temperature in 3 weeks	pH=2 After 1 week, the mixture is homogeneous when stirring. After 3 weeks, the mixture is homogeneous when stirring.	pH=3 The mixture is separated into 2 layers of water and emulsion after 1 week. The mixture is homogeneous when stirring. After 2 weeks, the mixture begins to separate the resin and water, settling the resin. After 3 weeks, the mixture completely separates alkyd resin and water.

The results from Table 6 showed that the AE2 alkyd emulsion resin (mixture of LAS and NP9 surfactants) began to show the phenomenon of separation after 2 weeks. After 3 weeks of storage at room temperature, the AE1 sample (mixture of SLS and NP9 surfactants) remained homogeneous and stable in the emulsion system.

Among the anionic surfactants, LAS and SLS, SLS emulsifiers naturally have Na⁺ ions that are well soluble in water, making them more stable with water than LAS. Using SLS, the stability of the emulsion is improved. The separation time of the mixture when using LAS is faster and less stable than when using the SLS emulsifier. Thus, the AE1 sample has better stability than the AE2 sample with the nonionic emulsifier NP9 and anionic SLS.

Stability of emulsion alkyd resin AE1 (SLS and NP9 surfactants mixture):

The stability of the emulsion alkyd resin was investigated by a storage method at room temperature (28±2°C), cold temperature (-5±2°C), and warm temperature (60±2°C). The stability of the alkyd emulsion resin AE1 is presented in Table 7 and Fig. 4.

Table 7. Stability of alkyd emulsion resin AE1.

Specifications	Alkyd emulsion resin AE1
State of the resin mixture	The mixture is liquid, milky white, stable, and homogeneous
Store at room temperature (28±2°C) for 21 days	The mixture is homogeneous and stable
Store at warm temperature (60±2°C) for 2 days	The mixture is homogeneous and stable
Cold storage at cold temperature (-5±2°C) for 01 day	The mixture is separated into 2 phases resin and water
Solid content (wt%)	52.8
Pre-storage pH	4
Post-storage pH	4
Ability to make coating films	Resin has ability to form coating films
Coating films	Coating films are thin, glossy, and smooth
Dried time (hard dried time)	48 h
Particle size at room temperature for 21 days and polydispersion (PI)	3601.1 nm and 0.834
Particle size stored at warm temperatures for 2 days and polydispersion (PI)	2771.4 nm and 0.926
Viscosity (25°C, seconds)	108

The results from Table 7 and Fig. 4 show the stability of alkyd emulsion resin AE1. After storing at room temperature for 21 days, the measured particle size was 3601.1 nm and the polydispersity index (PI) was 0.834 (Fig. 4A). Although the particle size of AE1 resin is quite large, the low dispersion indicates it is still stable [8]. After storage at warm temperatures, the AE1 resin size decreased to 2771.4 nm, and the dispersion increased to 0.926 (Fig. 4B). The mixture separated into two phases, resin and water, after storage at cold temperatures.

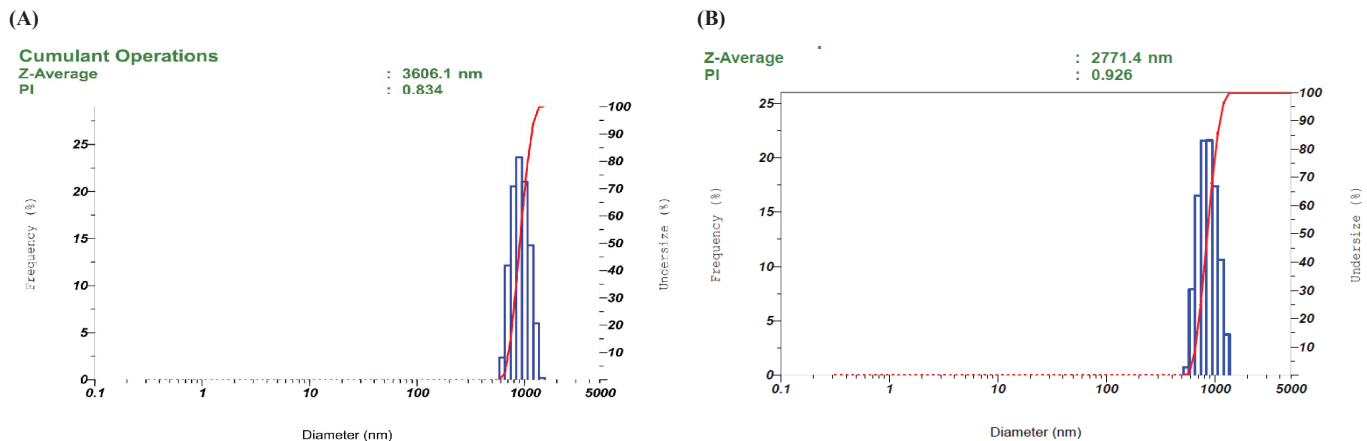


Fig 4. Particle size of emulsion alkyd resin AE1 with room temperature storage (A) and warm storage (B).

The decrease in particle size and increase in dispersion after warm storage can be explained as follows: micelles exist in the alkyd emulsion resin, meaning that alkyd resins are surrounded by two layers of surfactant. During the warm storage test, prolonged elevated temperatures cause the emulsifier to decompose from the surface and dissolve into the dispersed phase, thereby losing the emulsification effect. Consequently, the micelle size decreases when moisture is stored.

Alkyd emulsion resin AE1 remains in a liquid resin state and is suitable for application as water-based alkyd paint.

3.3. The physical chemistry properties and mechanical properties of water-based alkyd emulsion paint

The physicochemical properties and mechanical properties of water-based alkyd emulsion paint are presented in Table 8.

Table 8. Specifications of white alkyd emulsion paint.

Specifications	Standards	White alkyd emulsion paint (AEP)	Alkyd paint according to TCVN 5730-1993 standard
Fineness (µm)	TCVN 2091:2015	20	<23
Viscosity (s)	TCVN 2092:2013	130	>60
Coverage (g/m ²)	TCVN 2095:1993	56.9	<85
Adhesion	TCVN 2097:2015	1	<2
Flexibility (mm)	TCVN 2099:2013	1	<1
Pendulum hardness	TCVN 2098:2007	0.09	>0.15
Dried time (h)	TCVN 2096-3:2015	3.3	<22
Optical gloss (60°)	TCVN 2101:2016	48.1	>75
Impact strength (kg.cm)	TCVN 2100-2:2013	55	>45
Non-volatile content (%)	TCVN 10519:2014	65.5	>50

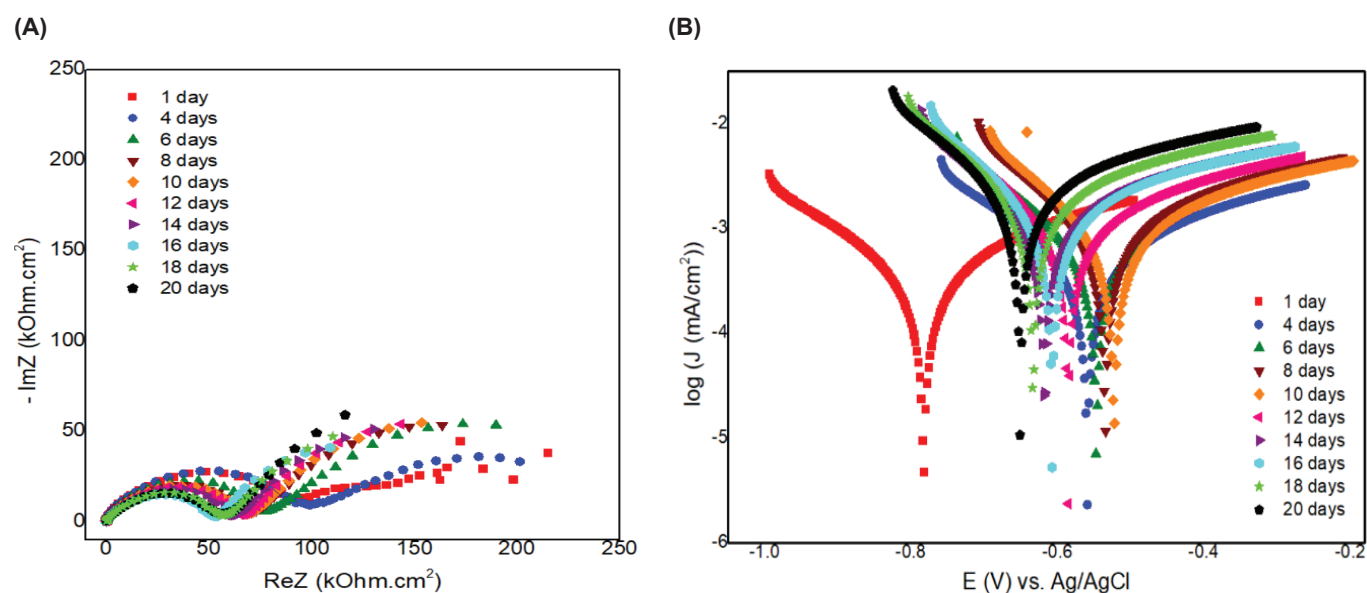


Fig. 5. Nyquist resistance diagram (A) and corrosion current density (B) of white emulsion alkyd paint sample.

The results from Table 6 show that AEP emulsion alkyd paint meets most of the characteristics of the paint in terms of fineness, coverage, viscosity, adhesion, flexibility, and impact strength according to TCVN 5730-1993 for alkyd paints. However, AEP alkyd emulsion paints with a water solvent will reduce the gloss and hardness of the paint film compared to solvent-based alkyd paints. However, using water as a solvent will significantly reduce the content of toxic volatiles.

3.4. Anti-corrosion properties of alkyd emulsion paints

The coating sample on steel was tested for electrochemical corrosion over a period of 28 days in a 3.5% NaCl solution. The results of the determination of Nyquist resistance and corrosion current density of the white emulsion alkyd paint sample are shown in Figs. 5 A and B, respectively.

From Fig. 5A, the Nyquist resistance spectrum of the AEP paint sample appears as a semicircle over time immersion in a 3.5% NaCl solution, which is characteristic of electrolyte immersion and steel corrosion. The coating corrodes rapidly over time. The polarization measurement from Fig. 5B also shows that the coating corrodes rapidly over time. This result is consistent with the Nyquist schema result in Fig. 5A.

AEP coating has good mechanical and physical properties. However, the corrosion resistance of the coating is still limited. Using aqueous solvents is an advantage of the paint, reducing the adverse environmental impact of

paints using organic solvents. However, alkyd emulsion paints need enhanced corrosion inhibitors and additives to increase shielding ability and improve the corrosion resistance of the paint film.

4. Conclusions

Alkyd resin was modified from soybean oil with a content of 50 wt%. The alkyd resin emulsion is stabilised with a mixture of SLS and NP9 with a suitable ratio of SLS:NP9 as 1:2 (wt/wt). The content of the emulsifier is 13 wt%. The ratio of alkyd resin to water is 50:50 (wt/wt), suitable for the alkyd resin emulsion process. Emulsion alkyd paint film has good mechanical properties that meet the standards for paint. However, the corrosion resistance of the paint film is limited and needs to be improved. The use of water solvents, gradually replacing organic solvents, holds great promise for environmentally friendly applications.

CRedit author statement

Le Huy Cuong Huynh: Methodology, Formal analysis, Original draft preparation, Visualisation; Khanh Xuan Phan: Conceptualisation, Data curation, Investigation; Thanh An Truong, Hung Thuy Nguyen: Investigation, Visualisation, Formal analysis.

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COMPETING INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this article.

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