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MECHANICAL PROPERTIES OF BIO-BASED SELF-HEALING COATING WITH EMBEDDED MICROCAPSULES

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Abstract

Self-healing coating is one of the smart coatings that can restore physical appearance and performance after exposure to damage such as scratches and weathering. Self-healing coatings have several benefits, including extending material life and lowering operating costs by reducing the need for planned maintenance, especially in situations involving remote locations. Microcapsule is one of the techniques that are widely used in the preparation of self-healing coating. This approach used an active healing agent to be stored in the microcapsule. In the previous work, pure jatropha oil (JO) and its derivatives known as jatropha oil-based polyurethane acrylate (JPUA) were encapsulated into a polyurea formaldehyde (PUF) microcapsule. In this study, JO and JPUA-IPDI-based microcapsules were loaded into the JPUA-TDI-based coating. 5 wt% and 10 wt% loading of microcapsules were dispersed using a mechanical stirrer in the coating formulation until a homogenous mixture was obtained. The self-healing coating formulation was coated on a steel plate (100 mm x 100 mm x 1.5 mm) using a bar applicator with a thickness of 50 µm,100 µm, 150 µm and 200 µm. Then, the wet film was irradiated under UV light until fully cured. Mechanical tests such as Pendulum Hardness Test (ASTM D4366), Cross-cut Adhesion Test (ASTM D3359-09), Transmittance Test (ASTM E1348), and Haze Test (ASTM D1003) were performed on the cured self-healing coating. The mechanical properties of the self-healing coating were compared with the coating without microcapsule loading as a control. It was found that the hardness value was reduced as the microcapsules were incorporated into the coating formulation in 5% JPUA and 10% JPUA. This trend continuously declined as the coating was thickened from 50 µm to 100 µm, 150 µm and 200 µm in the self-healing coating containing microcapsules. For the adhesion test, Control samples at all of the testing thicknesses showed a 4B adhesion score where less than 5% of the area was removed. A similar grade was displayed by 5% JPUA at 50 µm. The remaining samples indicated deteriorated adhesion properties as the film thickness increased and microcapsule loading increased. The 10% JPUA at a thickness of 200 µm showed the worst adhesion property with 15% to 35% of the removed area. As a conclusion, self-healing coating with 5 wt% loading of JO and JPUA-IPDI-based microcapsule with thickness of 50 µm has maintained the hardness, adhesion, haze, and transmittance properties when compared with the control sample.

Keywords: self-healing; microcapsule; mechanical properties; thickness; hardness

INTRODUCTION

The coating industry has witnessed a transformative shift with the advent of smart materials, particularly in the area of self-healing coatings. Self-healing coatings, also known as self-repairing coatings, have gained significant attention in recent years due to their ability to autonomously repair damage (Paquet et al., 2020). These coatings have the potential to revolutionize various industries, including marine, aerospace, and automotive, by increasing the durability and longevity of coated materials. By incorporating self-healing technology into coatings, materials can regain their structural integrity and functionality after being exposed to external forces or damage. This innovation solves the challenges faced by traditional coatings, such as limited product lifespan and susceptibility to corrosion (Liu et al., 2021). Furthermore, self-healing coatings have the potential to reduce maintenance and repair costs, as they can autonomously repair minor damages before they escalate into major issues that require costly repairs or replacements (Cheng et al., 2022). To develop effective self-healing coatings, researchers have employed various approaches such as microcapsule, vascular, and intrinsic systems. The microcapsule-based self-healing coating is one of the popular options and is nearly being commercialized (Ouarga et al., 2022). Via this approach, selective healing agent was encapsulated in a microcapsule followed by simply mixing procedure with the coating matrix. These microcapsules are designed to rupture upon damage such as scratch and crack on the coating surface. Subsequently, healing agents are released from the microcapsule to fill and repair the damages surface (Kothari & Iroh, 2023). The healing agents are hardened via polymerization process that induced either by catalyst or environmental factors such as UV light (Chen et al., 2019; Wang et al., 2019), oxygen (Song et al., 2022) and moisture(Alizadegan et al., 2018).

Currently, vegetable oils have been broadly used not only in food application but also in the industrial sectors such as paints, plasticizers, lubricants, and hardeners preparation and modification (Ataei et al., 2019; Karami et al., 2019). In order to reduce the carbon footprint, vegetable oils were also explored to be the healing agent (core content) for the microcapsule-based system. Tung (Li et al., 2018) and linseed (Çömlekçi & Ulutan, 2018; Wang & Zhou, 2018) oils are among popular oils that encapsulated directly without chemical modification. This is due to the high content of Iodine Value (IV) of these oils that allows them being polymerize when react with oxygen in the atmosphere (Li et al., 2018; Song et al., 2022). However, vegetable oils with low IV require chemical modification to tailor with the mechanism reaction during the repairing action. For example, palm (Saman et al., 2018) and coconut oil (Khorasani et al., 2017) have been reported to be converted to alkyd functionality before being encapsulated. Apart of that, Shisode and his team has reported the utilisation of pure soy oil with addition of cobalt drying agent (Shisode et al., 2018). To date, no study has been reported on utilisation of jatropha oil (JO) either in its pure or modified form for microcapsule-based self-healing coating purpose. JO is extracted from seed of *jatropha curcas* fruit. Apart of being locally available in Malaysia and South East Asia, JO (94 to 120 mg/g) has higher IV compared to palm oil (44 to 58 mg/g) that make it more reactive to chemical reaction particularly during the repairing mode (Amri et al., 2021).
Mechanical properties are crucial in determining the performance and durability of self- healing coating. However, the report on mechanical properties of microcapsule-based system is still limited to certain properties such as adhesion, hardness and bending and compression test (Jiang et al., 2021; Pongmuksuwan et al., 2023). For instance, a study on a microcapsule's wall made from melamine-formaldehyde was conducted whilst the core content was made from a mixture of carbonyl iron powder and multiwalled carbon nanotubes. The study revealed that as the addition of microcapsules increased, the gloss and adhesion of the coatings decreased. The addition of microcapsules to the coatings exhibited a tendency to increase the hardness, impact resistance and tensile properties, but followed by a decreasing trend (Wu et al., 2023). Despite the studies conducted in previous years,

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the scope of transmittance and haze properties on self-healing coating are still unexplored in existing studies. Therefore, it is important to study these properties especially when referring to self-healing coating that applied on glass and clear surface.

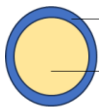
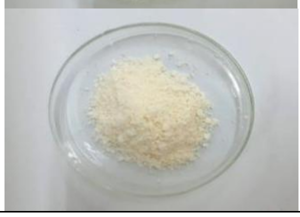
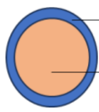
This work focuses on investigating the mechanical properties of a self-healing coating formulated with pure jatropha oil and the modified form; jatropha oil-based polyurethane acrylate as a healing agent into PUF microcapsule. The research examines key parameters such as microcapsule loading and coating thickness in relation to mechanical properties, including pendulum hardness, adhesion, transmittance, and haze. These mechanical properties are considered as a good guideline on product application and its durability.

MATERIALS AND METHOD

Materials

Pure jatropha oil was purchased from from Biofuel Bionas Malaysia Sdn. Bhd., Malaysia. Trimethylolpropane triacrylate (TMPTA) was procured from Sigma-Aldrich, Germany whilst benzophenone was supplied from Acros Organic (Belgium). Two series of jatropha polyurethane acrylate; JPUA-TDI and JPUA-IPDI were synthesized from crude jatropha oil and were based on different diisocyanate. The details of the procedure were explained in previous studies (Mudri et al., 2020, 2021). The microcapsules were prepared based on procedure reported by (Baharom et al., 2023). The properties of each microcapsule are tabulated in Table 1.

Table 1 Properties of JO 400 and IPDI 400

Microcapsule	Physical appearance	Schematic diagram
JO 400		
IPDI 400		

Preparation of Self-Healing Coating

Table 2 shows the components of the coating formulation for self-healing coating. Using the mechanical stirrer, JPUA-TDI, TMPTA and benzophenone were mixed until homogeneous. Subsequently, 5% and 10% of the microcapsules were dispersed in the coating formulation until a homogeneous mixture was obtained. Using a bar applicator, the film thickness was varied at 50 µm, 100 µm, 150 µm and 200 µm respectively on a glass plate (100 mm x 100 mm x 1.5 mm) and irradiated with UV light (UV-IST, Germany) for the curing process. Formulation without microcapsule was used as a control to determine the mechanical test for the self-healing coating. All films were then directly tested for their mechanical properties to determine the best combination of microcapsule loading and film thickness for the self-healing test.

Table 2 Components of self-healing coating formulation

	Self-healing coating			Microcapsule Loading	
Code	**JPUA-TDI (%)**	**TMPTA (%)**	**Benzophenone (%)**	**JO 400 (%)**	**IPDI 400 (%)**
Control	65	35	4	–	–
5% JO	65	35	4	5	–
10% JO	65	35	4	10	–
5% IPDI	65	35	4	–	5
10% IPDI	65	35	4	–	10

Mechanical Test

Pendulum Hardness Test

In accordance with ASTM D4366, the hardness of the UV-cured film was measured using a pendulum hardness tester (TQC, Netherlands) in Koenig damping mode. The readings (in seconds) were taken in triplicate for each sample during the damping time of the pendulum until the pendulum came to a complete stop. The average of the measured values was calculated.

Cross-Cut Adhesion Test

A cross-cut adhesion test (Biuged, China) was performed based on ASTM D3359-09. The purchased kit included a cutter blade, adhesive tape, brush and a magnifying glass. Based on the coating thickness for making the grid pattern, a 1 mm wide cutter blade was selected. The transparent 3M Scotch tape on the grid was peeled off at an angle of 180°. The brush was used to carefully remove the dirt and the magnifying glass was used to examine the surface.

Transmittance and Haze Tests

Haze Illuminant Tester (BYK-Gardner, Geretsried, Germany) was used to test the transmittance (ASTM E1348) and haze (ASTM D1003) for self-healing coating cured films. The tester measured both properties simultaneously as percentages. All measurements were performed in triplicate and the average value was calculated.

Morphology Observation

An optical microscope (Zeiss Primotech, Germany) was used to determine the morphology of the microcapsule shape in a coating film after UV curing.

RESULT AND DISCUSSION

Pendulum Hardness

Pendulum damping tests were performed to assess the flexibility and hardness of the coatings containing microcapsules, as presented in Figure 1. For Control, the hardness value has increased proportionally with the increasing thickness of the coating. The high number of oscillations reflects the good hardness but low flexibility in the Control coating. In radiation curing, acrylate functionality acts as active sites for crosslinking. The amount of acrylate functionality increased proportionally with the thickness of the film. Therefore, the hardness property increased as the possibility of crosslinking increased (Fu et al., 2021).

However, the hardness value was reduced as the microcapsules were incorporated into the coating formulation in 5% JO, 10% JO, 5% IPDI and 10% IPDI. This trend was continuously declined as the coating was thickened from 50 µm to 100 µm, 150 µm and 200 µm in each coating containing microcapsules. This is due to the possibility of the microcapsules interfering with the curing process. In this study, microcapsule was added via physical mixing where it did not have any chemical bonding with the polyurethane network in the coating. The compatibility of the PUF-based microcapsule in the coating can be improved either by using epoxy-based coating (Pongmuksuwan et al., 2023). In addition, Paquet et al., (2020)has demonstrated that introducing hydrogen bonding between the microcapsule shell and coating matrix has improved the hardness of the self-healing coating material. The film of 5% JO and 5% IPDI with 50 µm has comparable hardness performance with the Control (around 80 seconds) when tested at 50 µm thickness correspondingly.

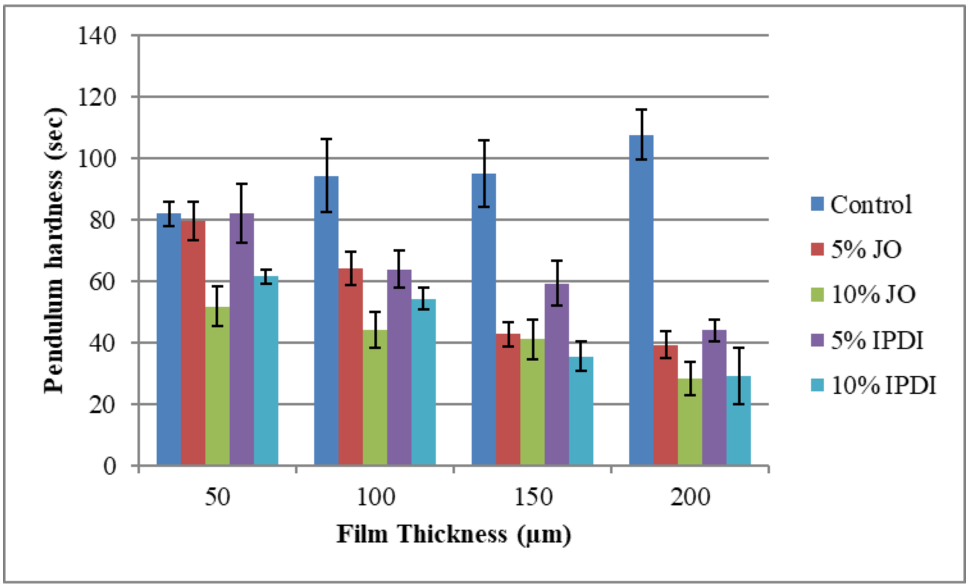


Figure 1: Pendulum hardness test for coating loaded with JO and JPUA-IPDI -based microcapsules

Adhesion Test

Cross-Cut Adhesion Test was conducted to investigate the effects of microcapsule embedment on the coating adhesion. The adhesion score of Control, 5% JO, 10% JO, 5% IPDI and 10% IPDI at thicknesses of 50 µm, 100 µm, 150 µm and 200 µm are tabulated in Table 3.

Table 3: Adhesion score of coating embedded with JO and JPUA-IPDI mixture-based microcapsules

Sample	Film Thickness (µm)	Adhesion Score
Control	50	4B
	100	4B
	150	4B
	200	4B
5% JO	50	4B
	100	3B
	150	3B
	200	3B
10% JO	50	3B
	100	3B
	150	2B
	200	2B
5% IPDI	50	4B
	100	3B
	150	3B
	200	3B
10% IPDI	50	3B
	100	3B
	150	2B
	200	2B

From Table 3, Control samples at all of the testing thicknesses showed a 4B adhesion score where less than 5% of the area was removed. A similar grade was displayed by 5% JO and 5% IPDI at 50 µm. The rest of the samples have deteriorated adhesion properties as the increased film thickness, and microcapsule loading increased. 10% JO and 10% IPDI at a thickness of 200 µm, respectively, showed the worst adhesion property with 15-35% of the removed area. The decline in mechanical properties in a coating with a high concentration of microcapsules might be due to non-uniform dispersion in the formulation. Therefore, the microcapsules also have restricted the mechanical bonding between the substrate and the coating matrix and caused poor performance in adhesion strength as the microcapsule loading was increased. Apart of that, the coating mixture becomes more viscous due to the additional loading of microcapsules. The high viscosity makes the coating formulation more difficult for to effectively penetrate and adhere to the substrate and led to the decrease in adhesion property (Pongmuksuwan et al., 2023). In this study, the formulation of 5% JO and 5% IPDI at a thickness of 50 µm were selected to be tested for self-healing test as the adhesion strength is comparable with the control where no microcapsule was added.

Transmittance Test

The effect of microcapsules loading at different film thicknesses is presented in Figure 2. All samples except 10% JO have a transmittance value of more than 85%, reflected a good transparency property (Adachi et al., 2018). This data indicated that the addition of microcapsules in 5% JO, 5% IPDI and 10% IPDI do not intervene in the transmittance property in the self-healing coating formulation.

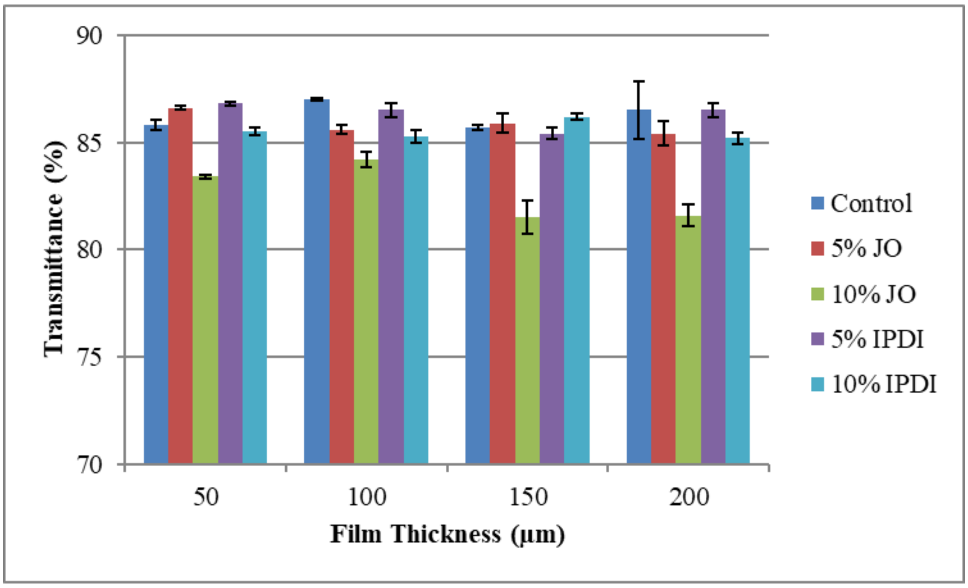


Figure 2 Transmittance value for jatropha oil-based self-healing coating with different thicknesses and concentrations of microcapsules

Haze Test

To examine the clarity of the film, a haze test was conducted. Figure 3 presents the haze property of self-healing coating containing microcapsules at different film thicknesses. The haze value increased as the film thickness increased in the for Control, 5% JO, 10% JO, 5% IPDI and 10% IPDI. Moreover, the increasing trend of haze was also observed with the increasing percentage of the microcapsule loading. This indicated that the higher film thickness and increasing number of microcapsules may cause poor dispersion in the coating formulation and led to surface roughness of the coating. The surface roughness is linked with poor haze (Adachi et al., 2018). Therefore, incorporating microcapsules is unsuitable for coating applications requiring transparent and clear surfaces such as glass, windows, and windscreens.

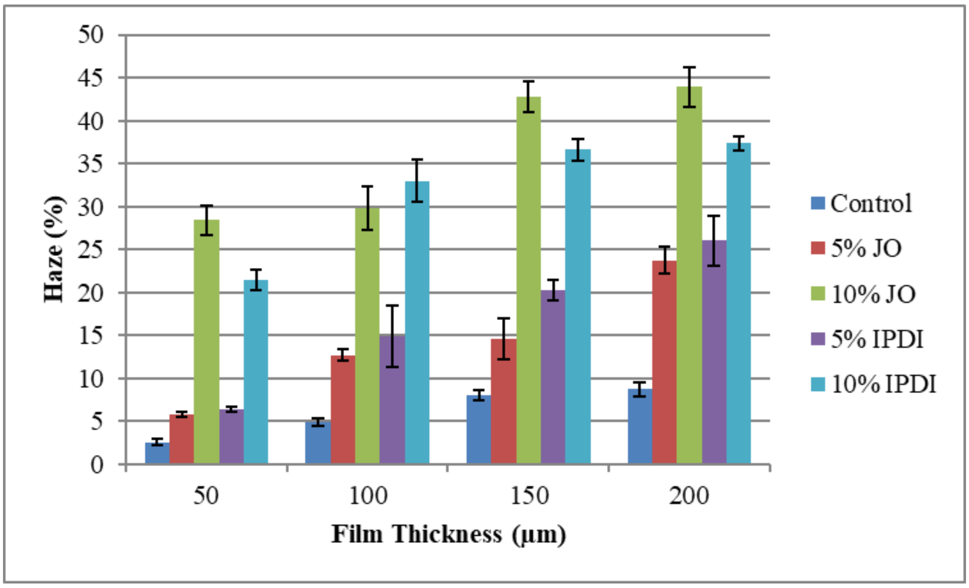


Figure 3 Haze property for jatropha oil-based self-healing coating at different film thicknesses and microcapsule loading

Morphology Observation

Considering all the mechanical tests that have been conducted, 5% JO and 5% IPDI with a thickness of 50 µm were selected to be evaluated for the self-healing test via scratch test in the future work. Figure 4 shows the overview of 5% JO and 5% IPDI coating with a thickness of 50 µm under an optical microscope at a magnification of 10x. It was found that both 5% JO and 5% IPDI coating have well-distributed microcapsule loading after being cured under UV light.

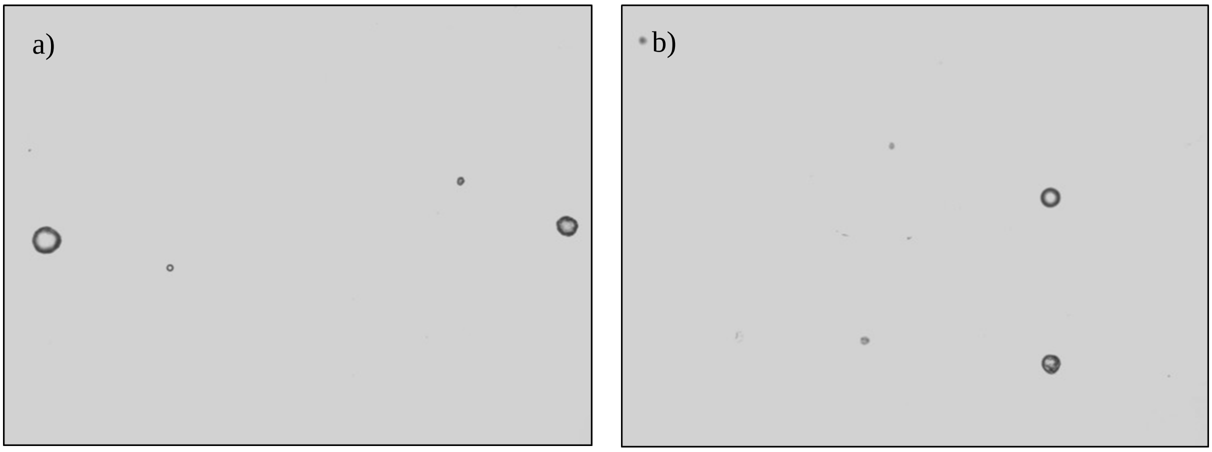


Figure 4 Optical micrograph of microcapsules distribution of a) 5% JO and b) 5% JPUA-IPDI at film thickness of 50 µm at 10x magnification after exposure to UV light

The observation was then zoomed in to 40x magnification to view the microcapsule morphology after being irradiated under UV light subjected to the curing process of the coating. From Figure 5, the microcapsules of both 5% JO and 5% IPDI were still intact, where no sign of PUF shell rupturing was detected when the microcapsule was irradiated under UV light. The thick and dark area was identified as the PUF shell whilst the core content was in the middle of the sphere with a light colour. This concluded that UV-curing technique is a suitable to be use for preparation of self-healing coating without any sign of damage on the microcapsule in the coating matrix.

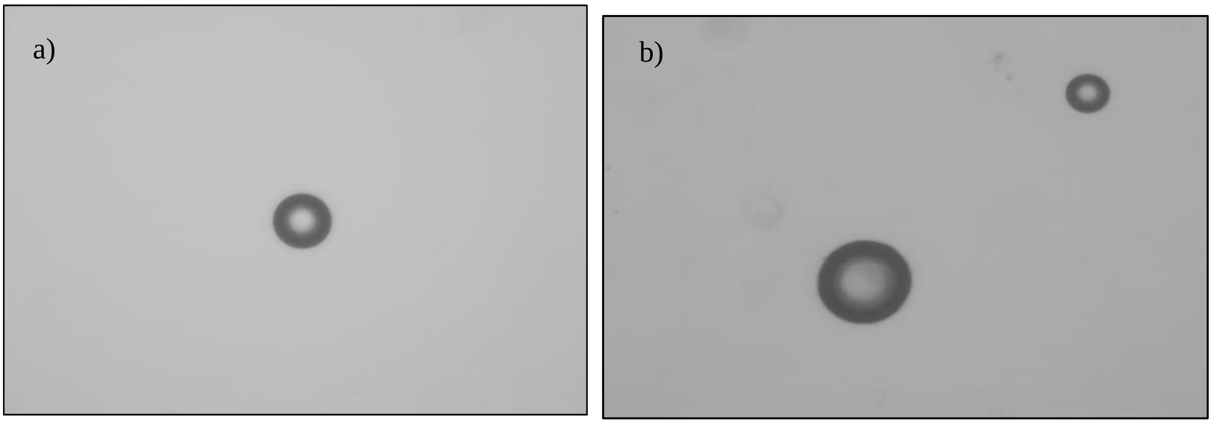


Figure 5 a) 5% JO and **b)** 5% IPDI under 40x magnification of an optical microscope after exposure to UV light at film thickness of 50 µm.

CONCLUSION

The addition of microcapsule loading and increasing coating thickness has declined the mechanical properties of the self-healing coating. The jatropha oil-based self-healing coating maintained their mechanical properties limited to 5 wt% microcapsule loading with film thickness of 50 µm when compared to the control sample. Under optical microscope, the 5% JO and 5% IPDI maintained spherical shape of the microcapsule after exposed to UV light prior to curing process where no sign of destruction was observed. A few recommendations are suggested for way forward to improve the mechanical properties such as (1) to study the microcapsule loading at below 5 wt%; (2) to prepare nano-size microcapsule; and (3) to examine the compatibility of the PUF microcapsule with epoxy-based coating.

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