1 Introduction

The solubility of inorganic substances/transition metal salts in a microemulsion is of great interest because of their outstanding scientific and industrial value. Various inorganic salts have been used as auxiliaries in the textile and dyeing industries for several centuries. Nowadays, indigo obtained from inorganic salts has not lost its importance because it offers better stability, especially oxidation resistance, compared with organic pigments. However, research on the solubility of inorganic compounds might play an essential role to unlock the physical and chemical aspects of thermodynamics and kinetics of the molecules microscopically, hence this understanding may be helpful for future advanced research, such as in nanotechnology, nanophotonics, nanocoatings, solar cell fabrication, membrane therapy, drug delivery to cancer cells, anti-counterfeiting material coatings, optical sensors, etc. In addition, the dissolution of inorganic pigments into microemulsions potentially has a bright future, as it can be applied in the pharmaceutical industry and in the conversion of light into energy, solar cells, sensor technology, and photonics.

Due to their polarity, inorganic pigments have better dissolution properties compare with their cousins called organic pigments. In aqueous solutions, inorganic pigments stick nicely in the water in the presence of surfactants. The head groups find it counters more quickly in the dispersion and are more dispersible than organic dyes. In w/o microemulsions, oil groups are surrounded by the cell-contained packs of inorganic pigments due to high water solubility. The thermodynamics solubility of inorganic pigments in the w/o microemulsions serve valuable information for physics and chemistry because such a phenomenon can be applied in nanoscale materials synthesis via nanoemulsions. This compact dispersion structure enables a long stability dispersion, allowing the production of long-lifespan printing inks and paints. The corresponding advantage of inorganic pigments is, of course, their resistance to oxidation and photolysis. In addition, it is also possible to synthesize nanomaterials with super electrical, magnetic, and optical properties.

Recently, yellow ink-jet ink nano-TiO2–Sb–Ni was prepared by Talavar, et al. using surfactant size control microemulsions.
The authors claimed that they could produce 65 nm size yellow nanoparticles of Ti/Sb pigment, used for ceramic tiles inks. Nano-TiO₂-Sb–Ni was first encapsulated in the w/o microemulsions to control the size then disassembling and redisperse the material in the surfactant/cosurfactant/water to fabricate the inks.²¹ Next, Wang, et al.²² synthesized Pr–ZrSiO₄ pigment via the sol–gel-microemulsion method, where ZrO₂ and SiO₂ sols are used as water phases, cyclohexane serves as oil phase, and Triton X-100 and 1-hexanol used as surfactant and co-surfactant, respectively. The results show that the materials were ready to be used as ink-jet ink due to the direct transformation of liquid/liquid to solid/liquid interface from sol to gels without emulsifiers’ assistance.²²

In this work, we would like to fabricate ballpoint ink from encapsulated inorganic pigments in the w/o microemulsions gel of Tween-60/cyclohexane/water. This research aims to understand the solubility properties of inorganic pigments in the w/o microemulsions gels and potential applications in the industries. This research can be implemented in chemistry, biology, pharmacy, food sciences, paint, and ink technology.

2 Material and methods

The materials used in this study were freshly prepared inorganic pigments/dyes such as Fe(CNS)₅ (red), Fe₂Fe(CN)₉ (blue), CdS (yellow), and CuS (black). Surfactant Tween-60 96% purity from Merck, cyclohexane 98% purity from Merck, double distilled water, Fe(NO₃)₃ 96% purity from Acros Chem., KSCN 98% purity from Merck, K₄[Fe(CN)₆] 98% purity from Merck, FeSO₄ 98% purity, HCl 36% pro analysys from Merck, CaCl₂ 98% from Acros Chem., CuSO₄ 98% from Merck, FeS 98% from Merck. All pigments were synthesized freshly in the laboratory using known procedures.

2.1 Pseudo-phase diagram of water-Tween-60/cyclohexane

Room temperature pseudo-phase diagram of oil in water and water in oil microemulsions (o/w-µE, & w/o-µE), lamellar liquid crystals (Lₐ), and hexagonal liquid crystals (H₃) determined by titrating cyclohexane (C₆) into the fixed composition of water (W) and Tween-60 (Tw-60) according to the relation 1:

\[ X_{\text{Tot}} = x_w + x_{Tw} + x_{C6} \]

Where \( X_{\text{Tot}} = 0.50 \) g, \( x_w = \) weight in % of water, \( x_{Tw} = \) weight in % of a surfactant, and \( x_{C6} = \) weight in % of cyclohexane.

Fig. 1 Molecular Structure of Tween-60 (Tw-60).

The phase boundary was determined carefully by visually investigating sample turbidity after vigorous stirring and shaking at room temperature (25–26 °C). Two-phase liquid with a clear boundary or cloudy mixture was grouped as coarse dispersion and discarded, whereas the clear, transparent, and translucent solution was grouped as microemulsions. Depending on the phase continuity and the position in the diagrams, the microemulsions were grouped as oil-in-water microemulsion (o/w-µE), and water-in-oil microemulsion (w/o-µE).

Next, to distinguish µE and liquid crystals, two paraffin plates were used to observe the light polarizability. The ability to rotate polarized light is the strong signature of liquid crystalline materials. There were two types of liquid crystals, namely lamellar/smectic liquid crystals (Lₐ) and hexagonal liquid crystals (H₃). To distinguish between Lₐ and H₃-phases, the materials were shaken strongly. The ability to move freely inside the tube was grouped as lamellar (Lₐ). It happened due to the layer structure of the lamellar. Where it has a two-dimensional degree of freedom by sliding along with the layer structures. Meanwhile, during shaking H₃-phase was stuck in the fixed position due to the compact structure. Therefore, the two phases were distinguished by observing the flexibility of materials in the shaking process.

2.2 Gelation process

TEOS and ethanol were added to a w/o microemulsion with a TEOS to water molar ratio of 1 : 8, then homogenized with a vortex mixer for about 15 minutes until the formation of sol. It was then heated in a water bath at 65 °C for 6 hours to form a gel.

2.3 Solubility studies of inorganic red and blue pigments in the gelation of water-in-oil microemulsion

The blue pigment was freshly prepared by taking the precipitate of Fe₃(FeCN)₉, resulting in the reaction of 50 ml of K₄[Fe(CN)₆] 1 M and 50 ml of FeSO₄ 2 M. The red pigment was prepared by adding 50 ml of Fe(NO₃)₃ 1 M to 50 ml of KSCN 3 M. Then, the red solution of Fe(CNS)₅ was collected.

The solubility test for the red and blue inorganic pigments was performed by adding each inorganic pigment sample to the w/o-µE. The addition was conducted by adding the inorganic pigment gradually until it could no longer be dissolved in the sample.

2.4 Solubility studies of inorganic yellow and black pigments in the gelation of water-in-oil microemulsion

The yellow pigment was prepared by reacting a 1 M solution of CaCl₂ and H₂S gas, where the H₂S gas was produced in a process in which FeS metal was mixed with 3 M HCl in Kipp’s apparatus. This reaction produced a yellow precipitate of CdS and was used as a yellow pigment. Meanwhile, to produce black pigments, a 1 M of CuSO₄ solution was reacted with H₂S. A black CuS precipitate was then produced and used as the black pigment.

The solubility test for inorganic yellow and black pigments was performed by adding each inorganic pigment sample to the
w/o-µE-gel. The addition was made by adding the inorganic pigment gradually until it could no longer be dissolved.

2.5 Density measurement
An empty 10 ml pycnometer was weighed before and after adding the gel sample and the pigments. Then, by calculating the mass of loaded gel divided by sample volume, we obtained the density of materials.

2.6 Refractive index measurement
An ABBE refractometer was used to measure the refractive index of the sample. Three sample drops were dropped onto the prism, and then the cap was closed. The light source (Na-lamp) was switched on, and the scale display was set to the refractive index of 1.30. Then the refractive index was measured for the desired sample (the sample’s refractive index is obtained when the light-dark image is at the top of the reading scale).

2.7 Turbidity measurement
The cuvette was cleaned and dried thoroughly before measurement, and 10 ml of the sample was added to the cuvette. The cuvette was inserted into the turbidimeter apparatus, and the measurements were repeated several times until the result was consistent.

2.8 Stability measurement and prototyping
The ink-encapsulated inorganic pigments in the w/o-µE-gel was put into the empty ballpoint-pen sleeves and used to write on the paper, and the physical state of the writing was monitored at regular intervals to determine the color fastness of the ink.

3 Result and discussion
First, the titration was used to construct the pseudo ternary phase diagram of water/Tween-60/cyclohexane. The construction of the pseudo ternary phase diagram followed the procedure of Friberg with slight modification. To obtain a pseudo ternary phase diagram of the µE, cyclohexane (C6) was added dropwise to Tw-60 and double-distilled (DD) water mixture and vortex-mixed until homogeneous. From visual observation, the transparent/one-phase liquid was considered as µE. By varying weight ratios, pseudo ternary phase diagrams were plotted, and the µE area was calculated using Origin® software. Calculating the µE area in the pseudo ternary graph was used to obtain the composition and the weight ratio of the components in the mixture. The area of µE was obtained and eqn (1) was used to obtain the percentage area in the pseudo ternary phase diagram.

3.1 Pseudo-phase diagram and phase area
Fig. 2 shows the phase diagram of the Tw-60/C6/water system. The µE area range extended along the composition line from 100% of C6 to 100% of Tw-60. The w/o-µE area was found in the shallow water content from 0% of water composition at 100% C6 to about 23% of water component at the weight fractions of water and Tw-60 line. The µE area started to form at 3% of Tw-60 and 1% of water components to 82% of Tw-60 and 27% of water components. The lamellar liquid crystal (Lα) area began to form at the Tw-60 composition containing 31% of Tw-60 and 11% of water components to a composition containing 81% of Tw-60 and 17% of water components, respectively. Hexagonal liquid crystals (Hα) area began to form at 21% of Tw-60 and 15% of water components to 79% of Tw-60 and 52% of water components. Even though Tw-60 was identified as a non-ionic surfactant, from Fig. 1. Molecular Structure of Tween-60 (Tw-60) could be inferred that the hydrophobic group in Tw-60 could be a stearate group with a longer carbon chain. This longer carbon chain allows Tw-60 to dissolve more onto nonpolar groups, making cyclohexane more soluble in Tw-60.

The complete description of the phase area of the Tw-60/C6/water system is well described in Fig. 2a.

In Fig. 2b, the sample areas were chosen to conduct the solubility test. The points were named S1, S2, and S3. All sample points have similar water compositions but different Tw-60 and
C6 compositions. In our experience, the gelation process was hardly determined by water composition rather than surfactant and co-surfactant (cyclohexane). Hence, representative of one dedicated sample point is enough to figure out essential information of the graph. Nevertheless, in the subsequent description, the sample point means S3, representing all sample points.

Phase determination was conducted by visual observation and also by paraffin. Physically, a microemulsion area was transparent with low viscosity, while an emulsion was cloudy. Paraffin was used to distinguish between liquid crystals from microemulsions and/or emulsions. Liquid crystal samples look brighter under paraffin, while microemulsion and emulsion samples look faint, as shown in Fig. 3. Paraffin acts as a polarizer and changes the direction of in-plane polarization of visible light.

Two paraffin were used for this purpose, acting as polarizers and analyzers. The incident light was passed through the sample, and the observer could show the brightness or color change from the other side. Liquid crystal materials show strong emissions which could be distinguished by the naked eye.

The two types of liquid crystals in the present work: are lamellar/smectic (L_a) and hexagonal liquid crystals (H_3). These two types of liquid crystals can be distinguished by their properties when stirred with a vortex mixer. L_a-liquid crystals have lower viscosity and can be stirred vigorously using a vortex mixer, while H_3-liquid crystals have a higher viscosity and cannot be stirred easily by using a vortex mixer. Due to this property, both liquid crystalline phase structures could be distinguished.

The sol–gel method was used for gelating w/o-µE. The sol–gel method prepares inorganic silica compounds by chemical reactions in the solution at low temperatures. The sol–gel method involves a phase change from a colloidal suspension (sol) to a continuous liquid phase. Sol is a suspension of colloidal particles (nanoparticles) that are stable in the liquid form. The process of sol formation occurs when TEOS is added to a w/o-µE to form a silica matrix during the gelation process. The interaction between the hydroxyl and ethoxy groups in TEOS was more robust and was challenging to break.

In this work, yellow pigment (CdS) was prepared from the reaction of CdCl_2 and H_2S precursors according to eqn (2):

\[
\text{CdCl}_2 + \text{H}_2\text{S} \rightarrow \text{CdS} + 2\text{HCl} \tag{2}
\]

The red pigment [Fe(SCN)_3]^2- was prepared from the reaction of Fe(NO_3)_3, and KSCN precursors according to eqn (3):

\[
\text{Fe(NO}_3)_3 + 3\text{KSCN} \rightarrow [\text{Fe(SCN)}_3]^2- + 3\text{KNO}_3 \tag{3}
\]

The blue pigment Fe_2[Fe(CN)_6] or Prussian blue was prepared from the FeSO_4 and K_4[Fe(CN)_6] precursors according to eqn (4):

\[
2\text{FeSO}_4 + 3\text{K}_4[\text{Fe(CN)}_6] \rightarrow 2\text{Fe}_2\text{Fe(CN)}_6 + 2\text{K}_2\text{SO}_4 \tag{4}
\]

The CuS (black pigment) was synthesized from the CuSO_4 precursor according to eqn (5):

\[
\text{CuSO}_4 + \text{H}_2\text{S} \rightarrow \text{CuS} + \text{H}_2\text{SO}_4 \tag{5}
\]

In our experiment, the best proportion of adding TEOS/ethanol to a w/o microemulsion was at a molar ratio of 1 : 8 because it gives optimal yields. The addition of TEOS and ethanol resulted in turbid, viscous, and denser structures compared with stand-alone precursors. The method started with sol formation, where the sol was mixed using a vortex mixer until the turbidity disappeared and the clear transparent liquid was formed.

The reaction continued with the gelation process when the silica matrix was formed when the ethoxy group of TEOS reacted with water molecules to form hydroxyl-substituted intermediates. TEOS was then hydrolysed by adding organic solvents such as ethanol to form silanol (Si-OH), followed by a condensation process in which the silanol group transformed into a siloxane group.

During gel formation, the initial TEOS concentration was inversely proportional to the size of the particles. The higher TEOS concentration leads to smaller particle size, reducing the solution density and increasing viscosity. A gel is a colloidal mixture with two distinct phases, solid and liquid—concentration and temperature influence the gel formation process. The gel was formed at 65 °C by hydrolysis and condensation of cross-links. The heating process lasts 6–12 hours and results in a semi-solid gel with a soft, chewy/rubbery, and transparent.25

3.2 Solubility studies of inorganic pigments in the microemulsion sols (µE-sol)

Solubility of inorganic pigments in the µE-sol can simply be summarised in Table 1.

Solubility of inorganic pigments (g) for black, yellow, red, and blue are 2.9 <S> 3.6 (mg g^-1). The highest solubility value is given by Fe(CNS)_3 (red pigment), whereas the lowest solubility is given by CdS (yellow pigment).

The solubility of inorganic pigments can be explained by the solubility of salts solution in the microemulsions.26 Solubility of [Fe(SCN)_3]^2- (red), Fe_2[Fe(CN)_6] (blue), and CuS (black) were relatively the same because the cation size of Fe^{3+} (78 pm), Fe^{3+} (69 pm), and Cu^{2+} (73 pm) of the salts are relatively the same. Whereas the solubility of CdS was relatively lower because the
cations. On the other hand, the lowest density, refractive index, and turbidity were given by yellow pigments. The result shows that the highest density, refractive index, and turbidity were given by red inorganic pigment as well as refractive index and turbidity. It could be explained; because the number of materials added to the system was higher, resulting in an increasing number of components. On the other hand, the lowest density, refractive index, and turbidity were given by yellow pigments. It was due to the quantity of added materials to the system being much lower.

The µE-gel’s physical appearance after pigment addition is presented in Fig. 4. Each pigment demonstrated good solubility in the µE-sol, and after homogeneously mixing, the microscopic mixture shows excellent stability.

### 3.3 Prototype design and stability

The ballpoint prototype design was illustrated in Fig. 5. Some quantities of encapsulated inorganic pigment in w/o-µE was inserted in the pen’s sleeve and used as writing ink. The results show that the inorganic pigment in µE-gel performs well, as well as a regular ink pen. For blue pigment, the writing shows a solid Prussian blue color. Although the time for solvent evaporation was a bit longer, compared with the commercial blue pen, in general, the result was satisfactory. The red, yellow, and black pigment demonstrated a solid color as well as Prussian blue, but the color stability was not as good as Prussian blue. Notably, the black ink gradually fainted after three days of exposure to air. Nevertheless, comprehensive research should be conducted to understand the inks’ stability properties fully.

### 4 Conclusions

The pseudo-phase diagram of Tween-60/cyclohexane/water was used to map w/o-microemulsion, lamellar/smectic liquid crystals, and hexagonal liquid crystals. A given composition sample of w/o microemulsion area was used to study the solubility of inorganic pigments [Fe(SCN)]3− (red), Fe2Fe(CN)6 (blue), CdS (yellow), and CuS (black). The result shows that the highest solubility value was given by Fe(CNS)3 (red pigment) of 3.65 ± 0.05 mg g−1, whereas the lowest solubility was given by CdS (yellow pigment) of 2.92 ± 0.05 mg g−1. Meanwhile, for Fe2Fe(CN)6 (blue), and CuS (black), the solubility was ~3.2 ± 0.05 mg g−1. The solubility difference is due to the cation and anion size, where the more extensive of the cation/anion, the bigger the hydrated radii, resulting in challenges to diluting in the microemulsion system. Furthermore, some quantities of pigments encapsulated in microemulsion gel have been

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**Table 1** Solubility, density, refractive indices, and turbidity of sol after being introduced by inorganic pigments

| Sample (S) | Maximum solubility (S) mg g⁻¹ | Density (ρ) g cm⁻³ | Refractive indices (n) | Turbidity (θ) NTU |
|------------|-------------------------------|-------------------|-----------------------|------------------|
| Black      | 3.22 ± 0.05                   | 0.924 ± 0.05      | 1.4331 ± 0.05         | 1.90 ± 0.05      |
| Yellow     | 2.92 ± 0.05                   | 0.915 ± 0.05      | 1.4225 ± 0.05         | 1.87 ± 0.05      |
| Red        | 3.63 ± 0.05                   | 0.960 ± 0.05      | 1.4360 ± 0.05         | 1.89 ± 0.05      |
| Blue       | 3.21 ± 0.05                   | 0.922 ± 0.05      | 1.4149 ± 0.05         | 1.85 ± 0.05      |

α: 0.82 g cm⁻³, n: 1.4074, θ: 1.80 NTU, according to Indonesian National Standard (SNI) No. 06-1567-1999, the density of commercial ink should be between 0.9–1.0 g cm⁻³ (Renggains et al., 2017). The uncertainty values is determined using 95% significance value for 3-sample points S₁, S₂, and S₃.

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![Fig. 5](image_url) The ink prototype of (a) blue, (b) red, (c) yellow, and (d) black.
inserted into the ballpoint sleeves as a prototype, and the demonstration shows that it could be used as a ballpoint ink. Finally, research on the stability of the ink under light, temperature, oxidation, and chemical need to be conducted more to understand the physico-chemical properties of inorganic salts solubility in microemulsion gels.

Author contributions

DB: conceptual – planning, lab experiments, writing – original draft, writing – review & editing, methodology, formal analysis; SB, NYS, FY, EE: lab experiment, working on characterization, AA: conceptual, writing – review & editing, methodology, formal analysis, visualization, project administration, & resources; GG: formal analysis, project administration, & resources.

Conflicts of interest

The authors have no conflicts to declare.

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