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Research article

Conversion of chromium from simulated batik waste through the utilization of gamma irradiation technique to produce potassium dichromate

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Abstract: Chromium (Cr(VI)) is a type of hazardous waste generated by the batik industry. In this study, the gamma irradiation technique was applied to precipitate chromium in simulated batik mordanting waste. Gamma irradiation induced the generation of active species, including hydrated electrons (e_{aq}) and hydrogen atoms (•H), which effectively reduced Cr(VI) ions to Cr(III) and caused precipitation as chromium hydroxide (Cr(OH)₃) upon interaction with hydroxide ions (OH⁻). Optimal precipitation conditions were observed at 40 kGy irradiation dose, 4M of 2-propanol work as scavenger, and pH 9. The applied reaction conditions led to a decrease in the concentration of Cr(VI) in simulated waste from 11,673 ppm to 177 ppm. The Cr(OH)₃ was calcined to form chromium oxide (Cr₂O₃) and through a chemical process was synthesized back into K₂Cr₂O₇ with the total Cr recovery of 63.39%. SEM and FTIR analysis indicated that the recovery of Cr(VI) into Cr(OH)₃, Cr₂O₃, and K₂Cr₂O₇ can be considered successful.

Keywords: potassium dichromate; batik waste; gamma irradiation; chromium

1. Introduction

Batik has developed and evolved throughout its extensive journey. Originating from the limited environment of the palace, batik has now evolved into one of Indonesia's creative industry commodities entering the global market. Batik is a motif technique that uses "canting" or stamps as its

drawing media, and the colors of batik itself are applied using dyes. There are two types of dye materials for batik: Natural dyes are extracted from natural substances and synthetic dyes, which are artificial dyes produced by factories [1]. Batik was officially recognized by UNESCO as a Masterpiece of the Oral and Intangible Heritage of Humanity on October 2, 2009 [2]. The formal recognition from international institutions regarding batik can be assumed to have a positive correlation with the increased production and consumer demand for batik. The batik industry has long been a sector that significantly contributes to the national economy.

In addition to the positive impacts it has brought, the development of the batik industry has also raised environmental issues due to the potential pollution from its waste. The waste, characterized by dark color and containing hazardous heavy metals, is generated during the batik process, which includes mordanting, waxing, dyeing, fixation, and the final stripping process [3]. Besides that, toxic heavy metal compounds found in the industrial waste of batik include chromium (Cr), lead (Pb), nickel (Ni), copper (Cu), and manganese (Mn) [4].

Chromium (Cr(VI)) was come from a process called mordanting. Mordanting was a process to overcome the limitation of natural dyes that were difficult to penetrate into the fabric and prone to fading. Mordanting is employed during dyeing, involving the use of mordant compounds to enhance the dye's affinity for the material/fabric [5]. Some effective and commonly used mordants include potassium aluminum sulfate (KAl(SO₄)₂); potassium dichromate (K₂Cr₂O₇); iron(II) sulfate (FeSO₄); copper(II) sulfate (CuSO₄); and tin(II) chloride (SnCl₂) [6].

Cr(VI) heavy metal is a persistent, highly bioaccumulative, and toxic chemical that does not easily degrade in the environment and accumulates in the body through the food chain[7]. The toxicity of chromium species depends significantly on its oxidation state. Chromium as Cr(VI) is toxic and considered carcinogenic, while chromium as Cr(III) is an essential micronutrient for humans[7]. The waste of Cr(VI) heavy metal is one of the hazardous wastes in the batik industry.

The heavy metal Cr(VI) in the liquid waste must be treated to become less hazardous before being safely disposed into the environment to reduce its toxicity by reducing it to Cr(III). Recent advancements in removal technique of Cr(VI) toxic ion from aqueous solution are adsorption, membrane technology, photocatalyst, electrochemical treatment, microbial treatment, microbial fuel cell, floatation, and ion exchange [8]. However, dealing with these methods has a fundamental disadvantage, namely the occurrence of secondary contamination in waste and the need for the use of reagents to separate deposits. Therefore, a new approach is needed to overcome these drawbacks, including utilizing the gamma irradiation technique.

When gamma rays hit a medium in the form of a solution, radiolysis reactions occur, producing radical species such as hydrated electrons (e_{aq}^{-}), hydrogen atoms ('H), and hydroxyl radicals ('OH) [9–11]. The bracketed values with reactive species produced from radiolysis of water depicts radiation chemical yield (G-value), the number of species (i.e., atoms, ions, and molecules) in micromole (μ mol) formed or consumed by 1 J of absorbed radiation energy[12]. The strong reducing effect of hydrated electrons (e_{aq}^{-}) and hydrogen atoms ('H) can directly reduce Cr(VI) ions to a lower valence state, turning them into Cr(III) that forms precipitate when reacting with hydroxide ions (OH⁻) present in wastewater as shown in the Eqs 1–4[13]. This reaction forms a Cr(OH)₃ precipitate, which can be separated by ordinary filtration.

$$H_2 O \rightarrow e_{aq}^- (0.28), \bullet H(0.06), \bullet OH(0.29), H_2(0.047), H_2 O_2(0.07), H_3 O^+(0.27)$$
 (1)

$$Cr^{6+} + 3 e_{aq}^{-} \to Cr^{3+}$$
 (2)

$$Cr^{6+} + 3 \bullet H \to Cr^{3+} + 3H^+$$
 (3)

$$Cr^{3+} + 30H^- \to Cr(0H)_3 \downarrow \tag{4}$$

The presence of hydroxyl radicals ('OH), which act as oxidizers, can interfere with the precipitation process, but their presence can be eliminated using a scavenger. Scavengers that can be used include 2-propanol, t-butanol, formic acid, and sodium formate [10,13]. The scavenger used in this study is 2-propanol.

The precipitation process of Cr with the radiolytic reduction method will be optimal in the pH range of 7–9.5. At a pH that is too low, hydrated electrons are quickly captured by hydrogen ions and converted into hydrogen atoms. On the other hand, at a pH above 9.5, it causes G-values for 'OH and e_{aq}⁻ decrease [10]. Additionally, Cr(III) compound will remain soluble at low pH and will begin to precipitate as Cr(OH)₃, reaching a maximum at pH 7 to 9.72 [14]. If Cr(OH)₃ is heated, Cr₂O₃ will be formed [9,13].

The difference between this research and the other research involving the reduction of Cr(VI) to Cr(III) is the uniqueness of batik waste composition. The batik waste contains dyes and wax that may influence the reduction and precipitation processes of chromium. In this research, the effect of irradiation doses, pH, and scavenger concentrations was studied to determine the optimal conditions for the formation of Cr precipitates from simulated liquid waste of batik using gamma irradiation. Furthermore, the formed Cr precipitates will be converted back into potassium dichromate through a chemical process so that the potassium dichromate can be reused. The percentage recovery of the resulting potassium dichromate from this recycling process was calculated.

2. Materials and methods

2.1. Materials

Jalawe fruit skin, batik wax, potassium dichromate (K₂Cr₂O₇) from Merck, pro analysis 2propanol from Merck, perchloric acid (HClO₄) from Supelco, sodium hydroxide (NaOH) from Merck, and aquadest from Merck Millipore with resistance 18.2 MOhm.cm were used in this study.

2.2. Instrumentation

A type I of gamma irradiator with a Co-60 radioactive source was used to irradiated sample. The irradiator was installed at Polytechnic Institute of Nuclear Technology BRIN with dose rate 2.930 kGy/h at November, 2022. The irradiator has been calibrated with reference from Riso High Dose Reference Laboratory, Technical University of Denmark. B3 DoseStix was used as dosimeter. Monitoring the pH solution was used a pH/C meter Hanna Instruments HI 2210, remained Cr(VI) concentration in sample solution was determined used XRF Rigaku Nex QC by generated standar curve of Cr(VI) from K₂Cr₂O₇. Analysis of solid samples resulted from precipitation was done with FTIR Shimadzu IR-Spirit and SEM-EDS Hitachi SU3500.

2.3. Procedures

2.3.1. Prepared simulated liquid batik mordanting waste (receipt from batik craftsmen in Giriloyo, Yogyakarta, Indonesia)

The 50 grams of Jalawe fruit skin in 500 ml of water was boiled for 2 hours, the filtrate was taken to get the natural dyes. The 3.3 grams of $K_2Cr_2O_7$ was added with 1 ml of natural dye from the extract of Jalawe fruit skin that has been made and 0.02 grams batik wax. The mixture was dissolved in 100 ml of water and stirred.

2.3.2. Separation of wax from waste solution

The liquid waste from batik fixation was filtered with Whatman paper to separate wax from the waste solution.

2.3.3. Effect of scavenger concentration on the Cr precipitation

The simulated waste was added with 2-propanol at variations of 1M, 2M, 4M, 6M, 8M, and irradiated at a dose of 40 kGy, as well as measured the pH of the waste. The obtained precipitate was dried in an oven at 105 °C for 2 hours, and the formed precipitate was weighed and the remained Cr(VI) at solution was measured.

2.3.4. Effect of irradiation dose on the Cr precipitation

The simulated waste was added with optimum scavenger concentration and irradiated at dose 10, 20, 30, 40, and 50 kGy and measured the pH of the waste. The obtained precipitate was dried in an oven at 105 $^{\circ}$ C for 2 hours. The formed precipitate was weighed and the remained Cr(VI) at solution was measured.

2.3.5. Effect of pH solution on the Cr precipitation

The simulated waste was varied the acidity level (pH) to 3, 5, 7, 8, 11, then added with optimum scavenger concentration and irradiated with optimum irradiation dose. The obtained precipitate is dried in an oven at 105 °C for 2 hours. The formed precipitate was weighed and the remained Cr(VI) at solution was measured.

2.3.6. Chromium recovery from simulated waste through gamma irradiation

The simulated waste was added with optimum scavenger concentration and conditioned at optimum pH, and Irradiated at optimum irradiation dose. The precipitate was filtered with Whatman paper and dried the precipitate at 105 °C for 2 hours. It was expected that $Cr(OH)_3$ is formed. Weighed the $Cr(OH)_3$ precipitate.

2.3.7. Synthesis of potassium dichromate [14]

The obtained $Cr(OH)_3$ precipitate was calcined at 500 °C for 5 hours. It was expected that Cr_2O_3 formed in this process. KOH was heated at 200 °C, after KOH melted completely, was added

 Cr_2O_3 with a mass ratio of KOH to Cr_2O_3 is 2:1. KNO₃ was added to the mixture with a mass ratio of KNO₃ to Cr_2O_3 is 3:1. The mixture was mixed until homogenous and becomes a thick paste.

The mixture was cooled and dissolved at 100 ml of aquadest then heated until dissolved. The solution was filtered and the obtained filtrate was collected in an evaporating dish (a yellow filtrate). The filtrate was heated at 120 °C until saturated, added glacial acetic acid dropwise while stirred (until it turned orange-red and did not change anymore). The filtrate was cooled immediately in an ice bath until crystals formed. The formed crystal was filtered then dried at 150 °C and weighed its mass. The percentage of chromium (Cr) recovery was calculated.

3. Results and discussion

3.1. Effect of scavenger concentration on the Cr precipitation

A scavenger is a radical attractor intentionally added to control the products of a reaction. In the context of the deposition of Cr(VI) into Cr(III) in the form $Cr(OH)_3$, the scavenger will attract •OH radicals thereby preventing the re-oxidation of Cr(III) back into Cr(VI) by reacting with •OH radicals. In this study, 2-propanol was employed as the scavenger. The 2-propanol reacts with •OH radicals (oxidative) and form reducing $(CH_3)_2C$ •OH radicals according to the reaction (6). Therefore, when adding a scavenger, the number of reducing radicals (G-value) in solution could be increased twice. Both solvated electrons and $(CH_3)_2C$ •OH radicals participate in reduction of Cr(VI) to Cr(III). The contribution of hydrogen radicals to this process is negligible, since their G-value is small.

The 2-propanol scavenger was varied with concentrations of 0 M, 1 M, 2 M, 4 M, 6 M, and 8 M. The measured pH of the waste solution before irradiation was 4.3 and the waste was irradiated 40.0 kGy. The graph showed that the lowest mass of $Cr(OH)_3$ precipitate was reached without the addition of 2-propanol scavenger. This occurs due to the absence of the radical scavenger, so that the hydroxyl ions (•OH) reacting with reduced Cr(III) ions. As a result, Cr(III) ions oxidize back to Cr(VI) ions without reacting with hydroxide ions (OH⁻) as shown at Eq 5.

$$Cr^{3+} + 30H \bullet \to Cr^{6+} + 30H^{-}$$
 (5)



Figure 1. The mass of the formed $Cr(OH)_3$ precipitate and the remaining concentration Cr(VI) in the solution as function of scavenger concentration.

The mass of the $Cr(OH)_3$ precipitate increases with the rising concentration of 2-propanol scavenger. The maximum amount of precipitate was observed at a concentration of 4 M followed by decreased Cr(VI) concentration in the solutions. At 6 M and beyond concentration of 2-propanol, the mass of $Cr(OH)_3$ is decreased. This indicates that the optimal capture of hydroxyl radicals (•OH) and hydrogen ions (•H) by 2-propanol occurs at a concentration of 4 M.

$$(CH_3)_2 CHOH + OH \bullet \rightarrow (CH_3)_2 C \bullet OH + H_2 O \tag{6}$$

$$(CH_3)_2 CHOH + H \bullet \to (CH_3)_2 C \bullet OH + H_2$$
(7)

Reactions (6-7) [15] result in the release of one hydrogen atom from 2-propanol, produced the unstable radical $(CH_3)_2C$ •OH. To achieve stability, this radical reacted with Cr(VI) ions to created Cr(III) and C₃H₆O (propanal) as the products (Eq 8). Furthermore, the formed Cr(III) ions reacted with hydroxide ions (OH⁻) to form the Cr(OH)₃ precipitate.

$$[3(CH_3)_2 C \bullet OH + Cr^{6+} \to Cr^{3+} + 3H^+ + 3C_3H_6O \tag{8}$$

Increasing the concentration of 2-propanol after 6 M will decrease the mass of $Cr(OH)_3$ precipitate. This occurs because the excessive concentration of 2-propanol scavenger competes with hexavalent chromium to react with hydrated electrons (e_{aq}). According to the calculation, the amount of scavenger needed capture the •OH and •H radicals are 0.14 M, with estimation of precipitated $Cr(OH)_3$ was 0.47 g.

3.2. Effect of irradiation dose on the Cr precipitation

The simulated chromium waste was irradiated with varying doses of 10 kGy, 20 kGy, 30 kGy, 40 kGy, and 50 kGy, with the addition of 4 M 2-propanol as scavenger and the measured pH before

irradiation was 4.3. Figure 2 showed that with increasing irradiation doses, the mass of the formed $Cr(OH)_3$ precipitate increased. The optimum mass of the $Cr(OH)_3$ precipitate was observed at dose 40 kGy, but followed by a decreased at dose 50 kGy.



Figure 2. The mass of the formed $Cr(OH)_3$ precipitate and the remaining Cr(VI) concentration in the solution as function of total irradiation dose.

The increase in the mass of the $Cr(OH)_3$ precipitate occurs because the amount of reducing agents produced is influenced by the absorbed dose. The higher the irradiation dose, the more reducing agents were generated from the radiolysis process. These agents reduced Cr(VI) ions to lower-charged chromium ions (Cr(III)). As a result, the concentration of Cr(VI) in the waste decreased and the formed $Cr(OH)_3$ precipitate increased.

At a dose 50 kGy, there is a decreased in the mass of the formed Cr(OH)₃ precipitate or an increase in the remaining Cr(VI) in the solution. This is due to the larger radiation intensity applied to the sample, causing more hydroxyl radical (•OH) compounds to be generated in the solution through water radiolysis. Consequently, the concentration of 2-propanol scavenger used should be higher. The concentration of the scavenger used is not sufficient to capture the abundant hydroxyl radicals formed at doses above 40 kGy. As a result, hydroxyl radicals (•OH) react with Cr(III) ions, and the Cr(III) ions are re-oxidized, producing Cr(VI) ions. This leads to an increased in the concentration of Cr(VI) in the waste solution, and the formed Cr(OH)₃ precipitate decreased.

3.3. Effect of pH solution on the Cr precipitation

The acidity levels of simulated chromium waste were varied to pH 2, 4, 7, 8, 9, 10, and 11 by adding sulfuric acid (H_2SO_4) or NaOH. The 4 M of 2-propanol as a scavenger was added, followed by irradiation at a dose of 40 kGy. Figure 3 showed the optimum formation of Cr(OH)₃ precipitate and the lowest remains of Cr(VI) was observed at alkaline pH, with the optimum pH was 9. Some studies

have reported that the optimum pH for this conditions is between 8.5–9.5 due to the solubility of chromium hydroxide in that range [16,17]. It is rational to expect that the generated Cr(III) could be spontaneously precipitated under alkaline conditions due to the low solubility of $Cr(OH)_3$ (pKsp = 30.2) [17]. However, it dissolves more readily in low or acidic pH conditions. $Cr(OH)_3$ is amphoterous, that can react with both hydrogen and hydroxide ions.



Figure 3. The mass of the formed $Cr(OH)_3$ precipitate and the remaining Cr(VI) concentration in the solution as function of pH.

At pH 2 and 4, which are acidic conditions, an excess of hydrogen ions (H⁺) was formed, leading to more reactions between hydrogen and hydroxide ions than the precipitation reaction. At pH 8 and the optimal pH of 9, which are alkaline conditions, the formation of an excess of hydrogen ions (H⁺) was minimized. This ensures that the precipitation reaction was not disturbed by the reaction between hydrogen and hydroxide ions, facilitating the desired $Cr(OH)_3$ precipitation. At pH 7 (neutral conditions), the concentration of hydrogen ions (H⁺) and hydroxide ions (OH⁻) was balanced, resulting in no excessive reaction between H⁺ and OH⁻, as observed in acidic and alkaline environments. In this condition, $Cr(OH)_3$ precipitation can occur but not optimal. At pH 11 (highly alkaline conditions), if there is an excess of OH⁻, the Cr(OH)₃ precipitate will redissolve, forming tetrahydroxo chromate (III) ions with the following reaction equation:

$$Cr(OH)_3 + OH^- \rightleftharpoons [Cr(OH)_4]^- \tag{9}$$

3.4. Chromium recovery from simulated wastewater treated with gamma irradiation

The optimum conditions for chromium precipitation from the simulated batik fixation waste were at scavenger concentration of 4 M, irradiation dose of 40 kGy, and at alkaline environment with a pH

before irradiation was 9. The resulting $Cr(OH)_3$ precipitate under these conditions was 1.678 grams from 3.3 grams of K₂Cr₂O₇ in the simulated waste. The Chromium recovery in this process was 72,6%.

Chromium(III) Oxide (Cr₂O3) formation occurs through the calcined of chromium(III) hydroxide $Cr(OH)_3$ at a temperature of 500 °C for 5 hours as shown at Eq10. From 1.678 grams of $Cr(OH)_3$ was obtained 1.2 grams of Cr_2O_3 . The Chromium recovery in this process was 96.9 %.

$$2Cr(0H)_3 \to Cr_2O_3 + 3H_2O$$
 (10)

By Heating a mixture of KOH, KNO₃, and Cr_2O_3 was produced a yellow filtrate, namely potassium chromate (K₂CrO₄). The reaction formed at Eq 11 [18].

$$Cr_2O_3 + 3KNO_3 + 4KOH \rightarrow 2K_2Cr_2O_4 + 3KNO_2 + 2H_2O$$
 (11)

The acidification process was carried out by adding glacial acetic acid to convert potassium chromate into orange-colored potassium dichromate ($K_2Cr_2O_7$). Potassium dichromate was stable in an acidic environment and the ion's reaction was reversible. In a neutral or alkaline solution, chromate ions are stable, while in an acidic environment, chromate ions are present. Besides accelerating the formation of potassium dichromate, the addition of acetic acid aims to bind impurities that may be present in the potassium dichromate formation process. The reaction that occurs was shown at Eq 12 [18].

$$2K_2CrO_4 + 2CH_3COOH \rightleftharpoons K_2Cr_2O_7 + 2CH_3COOK + H_2O$$
(12)

Subsequently, the purification of the resulting potassium dichromate product is carried out using the recrystallization principle, where the substance is cooled directly in an ice bath while in a hot state. This accelerates crystal formation. The next steps involve filtration and drying of the crystals. The mass of potassium dichromate ($K_2Cr_2O_7$) formed in this process was 2.01 grams from 1.2 grams of Cr_2O_3 . The Chromium recovery in this process was 89.28 %, with the total Cr recovery was 63.39%.

3.5. SEM characterizations of $Cr(OH)_3$, Cr_2O_3 , and $K_2Cr_2O_7$ obtained from simulated wastewater recovery

The SEM results of $Cr(OH)_3$ (Figure 4a) showed a rough surface at a magnification of 2000 times. This aligned with previous study which states that at the micrometer scale $Cr(OH)_3$ SEM results showed a rougher surface compared to Cr_2O_3 [19]. The C_2O_3 (Figure 4b) particles appear smaller than $Cr(OH)_3$ and showed concave pits indicating water loss in the Cr_2O_3 formation through calcination. SEM results for Cr_2O_3 showed that all particles exhibit a spherical shape with a high level of agglomeration among fine particles. Potassium dichromate crystals (Figure 4c) appear as fine particles or particle clusters.



Figure 4. SEM Image 20000x of (a) $Cr(OH)_3$, (b) Cr_2O_3 , and (c) $K_2Cr_2O_7$.

3.6. FTIR characterizations of $Cr(OH)_3$, Cr_2O_3 , and $K_2Cr_2O_7$ obtained from simulated wastewater recovery

Figure 5 (a) showed the FTIR spectrum of the $Cr(OH)_3$ precipitate sample. An absorption band at around 940 cm⁻¹ was assigned to stretching vibrations of the Cr-O bond [20]. The two weak and broad bands at about 1630 cm⁻¹ and 3400 cm⁻¹ are due to the O-H vibration of water molecules adsorbed on

the surfaces [21]. The peaks at 1480 cm⁻¹ and 1370 cm⁻¹ can be assigned to the splitting of the asymmetric stretching of metal carbonate [22] absorbed from the air during the synthesis.

Figure 5(b) presented the FTIR spectrum of the Cr_2O_3 sample calcined at 500 °C. The result aligned with previous research which showed two sharp peaks at 575 and 630 cm⁻¹ assigned to the Cr-O stretching [21]. The peak at 3416 cm⁻¹ is attributed to the stretching of O-H from hydroxyl groups bonded with H. Peaks at 1039 cm⁻¹ and 1628 cm⁻¹ represent the bending vibration of H-O-H from physically adsorbed water molecules. The peak at 950 cm⁻¹ is associated with the stretching of Cr-O bonds.

Figure 5 (c) presented the FTIR spectrum of the $K_2Cr_2O_7$ sample and commercial one. The result showed peak at 555 and 754 cm⁻¹ confirming the previous studies. The peak at the wavenumber of 555 cm⁻¹ was caused by the symmetric stretching vibration of Cr-O-Cr, while the peak at 754 cm⁻¹ was caused by the anti-symmetric stretching vibration of Cr-O-Cr [23]. The peak at the wavenumber of 891 cm⁻¹ was attributed to the symmetric stretching vibration of Cr-O₃, while the very intense peak at 935 cm⁻¹ was caused by the asymmetric stretching vibration of Cr-O₃ [23]. It can be observed that there was no significant difference between the peaks of the K₂Cr₂O₇ sample and the K₂Cr₂O₇ commercial product. This indicated that the recovery of Cr(VI) into K₂Cr₂O₇ can be considered successful.



(a)



Figure 5. FTIR spectrum of (a) Cr(OH)₃, (b) Cr₂O₃, and (c) K₂Cr₂O₇ sample and commercial

4. Conclusions

The reduction of Cr(VI) to Cr(III) was carried in the synthetic chrome mordanting batik waste by gamma radiolysis of water in the presence of natural jalawe dye and the wax. Optimal precipitation conditions were observed at 40 kGy irradiation dose, 4M of 2-propanol work as scavenger, and pH 9. Radiolytic precipitation produced $Cr(OH)_3$, followed by calcination to form Cr_2O_3 . Through a chemical process, Cr_2O_3 can be change back to $K_2Cr_2O_7$. The SEM and FTIR result revealed that the recovery of Cr(VI) into $Cr(OH)_3$, Cr_2O_3 , and $K_2Cr_2O_7$ can be considered successful.

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Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The authors declare that they have no personal interest that could have appeared to influence the work reported in this paper.

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