

## Case Report

## The kinetics of the epoxidation reaction of sunflower oil utilizing peroxyformic acid generated in situ

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## ABSTRACT

Sunflower oil, rich in unsaturated fatty acids, is a promising renewable resource for bioplasticizers and biopolyols. While extensive research has been conducted on vegetable oil epoxidation, the kinetics of sunflower oil epoxidation using in situ-generated peroxyformic acid remains insufficiently explored. Most studies focus on peroxyacetic acid, leaving a gap in understanding the reaction kinetics and key parameters for peroxyformic acid epoxidation. This study investigates the kinetics of sunflower oil epoxidation using in situ-generated peroxyformic acid with a sulfuric acid catalyst. The effects of reaction temperature, time, and the molar ratio of double bonds to hydrogen peroxide were examined. A kinetic model was developed, and key parameters were determined, including reaction rate constants and activation energy. Results showed that temperature, reaction time, and reactant ratios significantly influenced iodine and oxirane conversions. While higher temperatures and prolonged reaction times initially increased oxirane conversion, excessive heat led to side reactions, reducing efficiency. The optimal conditions for epoxidation were found to be 60 °C, a reaction time of 3 hours, and a 1:2.0 molar ratio of double bonds to hydrogen peroxide, yielding the highest oxirane content. The reaction rate constants ranged from 7.7 to  $14.6 \times 10^{-6}$  liter/mol·s, with an activation energy of 19.4 kJ/mol. These findings provide critical insights into optimizing sunflower oil epoxidation, facilitating its potential use in sustainable biopolyol and bioplasticizer production.

## 1. Introduction

Double bonds in vegetable oils and fats have inspired extensive research into their derivatives for various applications, particularly concerning petroleum supplies' uncertainties. This condition has led to an increased focus on the utilization of derivative vegetable oils in the chemical industry, as these oils represent renewable resources that can be sourced from a range of crops and used to manufacture diverse products [1]. Global production of vegetable oils reached approximately 210.3 million metric tons in 2022–2023 and is expected to exceed 217 million metric tons in 2023–2024 [2]. Double bonds in vegetable oils can undergo chemical or enzymatic conversion to create epoxidized oils. These oils are extensively used across various industrial applications, including adhesives, coatings, elastomers, resins, composites, lubricants, bioplasticizers, and as feedstock for polyol production [3–5]. Consequently, global demand for epoxidized oils derived from vegetable oils is expected to rise annually.

Sunflower oil distinguishes itself among various vegetable oils due to its high concentration of unsaturated fatty acids, making it an excellent candidate for chemical modifications such as epoxidation [5–7]. The epoxidation process involves converting carbon-carbon double bonds (C=C) in the fatty acid chains into oxirane rings, which holds significant industrial relevance [8]. This process can be carried out using a variety of oxidizing agents, with peroxy acids being among the most commonly used options. Peroxyformic acid and peroxyacetic acid are notable for their ability to be generated in situ through reactions involving formic or acetic acid with hydrogen peroxide [9–12]. Utilizing peroxy acids produced in situ offers several advantages, including reduced handling of hazardous chemicals and enhanced control over reaction conditions. In industrial settings, epoxidized oil is typically produced by reacting peroxyformic or peroxyacetic acid with vegetable oils, often using a homogeneous sulfuric acid catalyst. However, heterogeneous catalysts derived from cationic ion exchange resins are also employed on a smaller scale. The peroxy acids required for the epoxidation reaction are

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generally generated in situ by reacting hydrogen peroxide with formic or acetic acid [9]. The main benefits of using homogeneous catalysts for the epoxidation of vegetable oils include a relatively high process yield, lower costs associated with the synthesis of peroxy acids, the potential for recovering carboxylic acid, and the easy accessibility of these acids [13].

Numerous studies have investigated the epoxidation of sunflower oil and various other vegetable oils [5,6,11,12,14–17]. However, the kinetics model and the effects of process variables related to the epoxidation of sunflower oil using in situ-generated peroxyformic acid have not been thoroughly examined. This research employs in situ-generated peroxyformic acid due to its greater reactivity than peroxyacetic acid. Gaining a comprehensive understanding of the kinetics model and the impact of process variables is crucial for optimizing reaction conditions and enhancing conversion to oxirane efficiency. The epoxidation reactions are affected by several factors, including temperature, reaction time, and molar ratios of the reactants. Analyzing these variables offers valuable insights into the kinetic models for the epoxidation process and opens up opportunities for improving scalability and cost-effectiveness, particularly in large-scale operations.

While numerous studies have investigated the epoxidation reaction using various vegetable oils and sunflower oil using various oxygen carriers, such as peroxyformic acid and peroxyacetic acid, the specific kinetics behavior of sunflower oil epoxidation with in situ generated peroxyformic acid remains insufficiently explored. Hence, this research aims to analyze systematically the kinetics of this reaction, determine rate constants, and evaluate the impact of the process variables, including temperature, reaction time, and the molar ratio of reactants such as double bonds and hydrogen peroxide, on iodine conversion and conversion to oxirane. The anticipated findings are expected to enhance the development of more efficient large-scale processes for producing epoxidized sunflower oil, offering potential applications across various industries focusing on sustainable and bio-based products. In addition, it will obtain model and kinetic parameters for peroxyformic acid generated in situ for sunflower oil epoxidation. The findings are expected to contribute to developing more efficient processes for producing epoxidized sunflower oil on a large scale, with potential applications in various industries focusing on sustainable and bio-based products.

## 2. Materials and methods

### 2.1. Materials

Sunflower oil was procured from a supermarket in Surabaya-Indonesia, while other additional chemicals, including methanol (Catalogue No 106009), 95–97 % sulfuric acid (Catalogue No 100731), sodium hydroxide (Catalogue No 106462), 30 % hydrogen peroxide (Catalogue No 107209), and formic acid (Catalogue No 100264) were supplied by Merck. Sodium sulfate anhydrous was provided by Smartlab. It is noteworthy that these substances were utilized without the need for further purification, ensuring both efficiency and convenience in the process.

### 2.2. Experimental setup

The epoxidation reaction was conducted in a three-neck round-bottom flask equipped with a thermometer, condenser, and mechanical stirrer. The flask was immersed in a water bath, allowing the temperature to be within a range of  $\pm 1$  °C for optimal results with higher oxirane conversion.

### 2.3. Characterization of sunflower oil

The fatty acid profile of sunflower oil was determined by converting it into Fatty Acid Methyl Esters (FAME) through derivatization using a sulfuric acid catalyst [18]. The modified procedure is as follows: 25 g of

sunflower oil, 5 ml of sulfuric acid catalyst, and 100 ml of methanol were added to a round-bottom flask that was immersed in a water bath. The mixture was then refluxed at 65 °C for 10 hours. Upon completion of the reaction, the product was allowed to cool and then neutralized with sodium hydroxide. Water was added to separate organic and aqueous phases. The organic phase was washed with warm water until achieving a neutral pH. Anhydrous sodium sulfate was added to the organic phase to eliminate residual water, and the mixture was filtered to yield dry oil. Finally, the oil phase containing FAME was analyzed using Gas Chromatography (GC), resulting in an FAME profile that determined the fatty acid content of the sunflower oil.

### 2.4. Epoxidation reaction

A specific quantity of sunflower oil, formic acid, and sulfuric acid was added to the round-bottom flask. While stirring the mixture, 30 % hydrogen peroxide was introduced drop by drop to prevent overheating, as the epoxidation reaction is highly exothermic. The reaction was carried out at various fixed temperatures and with different molar ratios of double bonds to hydrogen peroxide. Samples were collected hourly, and the reaction mixture was cooled before being neutralized with sodium hydroxide solution. Decantation was performed to separate the organic-soluble compounds (epoxidized oil) from the water-soluble ones. The epoxidized oil was subsequently washed with warm water in small aliquots until the washing water reached a neutral pH. Following that, sodium sulfate anhydrous was introduced to effectively reduce the water content, after which the mixture was filtered. Subsequently, the final product was subjected to analysis of its iodine value and oxirane content.

### 2.5. Analytical techniques

The iodine value was measured using the Wijs method [19]. A decrease in the amount of double bonds indicated the extent of reaction conversion, which was then calculated from the iodine value using the following equation [20]:

$$\text{Iodine Value Conversion (IVC)} = \frac{IV_0 - IV}{IV_0} \times 100\% \quad (1)$$

where  $IV_0$  represents the initial iodine value (g  $I_2$ /100 g oil) and  $IV$  is the iodine value at the designated reaction time (g  $I_2$ /100 g oil).

In the direct method, the oxirane oxygen content from experiments was determined utilizing a hydrochloric acid solution in diethyl ether (%  $OOC_{Exp}$ ) [19]. To calculate the theoretical maximum oxirane content (%  $OOC_{The}$ ), the following formula is employed [11]:

$$\%OOC_{The} = \left\{ \frac{\left( \frac{IV_0}{2A_i} \right)}{\left( 100 + \left( \frac{IV_0}{2A_i} \right) A_o \right)} \right\} \times A_o \times 100 \quad (2)$$

where  $A_i$  represents the atomic weight of iodine (126.9),  $A_o$  denotes the atomic weight of oxygen (16), and  $IV_0$  stands for the initial iodine value of the oil. Based on this research, the theoretical maximum oxirane content that could be achieved, as indicated by the initial iodine value, is 7.77 %. The conversion of double bonds to oxirane (RCO) was determined using the equation below [16]:

$$\text{Percentage conversion to oxirane (RCO)} = \frac{\%OOC_{Exp}}{\%OOC_{The}} \times 100\% \quad (3)$$

## 3. Results and discussions

The initial iodine value of sunflower oil utilized in this study was 133.7 g  $I_2$ /100 g oil. The fatty acid composition of the initial sunflower oil was determined through a derivatization method. In this process,

triglycerides in the oil were reacted with methanol in the presence of an acid catalyst to produce FAME, ensuring a complete reaction. The resulting (FAME) products were subsequently analyzed using Gas Chromatography (GC), and the obtained FAME composition was converted to reflect the composition of fatty acids. Table 1 presents the composition of the fatty acids in sunflower oil.

The fatty acid composition indicated a notable presence of unsaturated double bonds in sunflower oil, primarily attributed to oleic and linoleic acids, representing about 92.07 %. The characteristics of unsaturated fatty acids oleic and linoleic acids obtained in this research are similar compared to sunflower varieties in Brazilian with oleic acid in the range 14.0–39.4 % and linoleic acid in the range 48.3–74.0 % [21]. This characteristic positions sunflower oil as a potentially advantageous feedstock for producing epoxidized oil.

The epoxidation reactions were conducted under the following variable parameters: reaction temperatures ranging from 40 to 70 °C, reaction time between 0 and 8 hours, a molar ratio of double bonds (DB) to hydrogen peroxide between 1:1.5 and 1:2.5 (mol per mol), a molar ratio of DB to formic acid at 1:1 (mol per mol), and a catalyst loading of 0.5 % sulfuric acid.

### 3.1. Effect of reaction temperature and time

The impact of reaction temperature and time on the conversion of iodine value using in situ-generated peroxyformic acid is illustrated in Fig. 1. The results indicate that as the reaction temperature and time increase, iodine conversion is enhanced. Notably, a temperature of 70 °C demonstrates superior performance compared to lower temperatures, indicated by higher iodine conversion than other temperatures. Hence, a temperature of 70 °C is the optimal condition for iodine conversion. This trend suggests that double bond amounts decrease with rising temperature and time, reflected by a lower iodine value. A similar pattern was observed during the epoxidation of castor oil, where the iodine value diminished with increasing temperature and reaction time [12]. In the epoxidation process, double bonds are converted into oxirane rings. According to Arrhenius's Law, the reaction constant is temperature-dependent. Thus, higher temperatures result in an increased reaction constant, accelerating the reaction rate and improving iodine conversion [22].

Fig. 2 illustrates the changes in oxirane content indicated by conversion to oxirane over time at various temperatures. Conversion to oxirane increases as the reaction temperature and time increase at lower temperatures of 40 °C and 50 °C. The primary reaction, which involves converting double bonds into oxirane rings, becomes more pronounced, resulting in a higher conversion of double bonds to oxirane rings. At a higher reaction temperature of 60 °C, the conversion to oxirane peaks at 79.6 % after 3 hours. This peak occurs as the primary reaction competes with side reactions, with both temperature and time escalating. A similar pattern is observed in the epoxidation reaction at 70 °C, where the maximum conversion to oxirane occurs at 2 hours. However, the conversion to oxirane at this temperature remains lower than the peak observed at 60 °C.

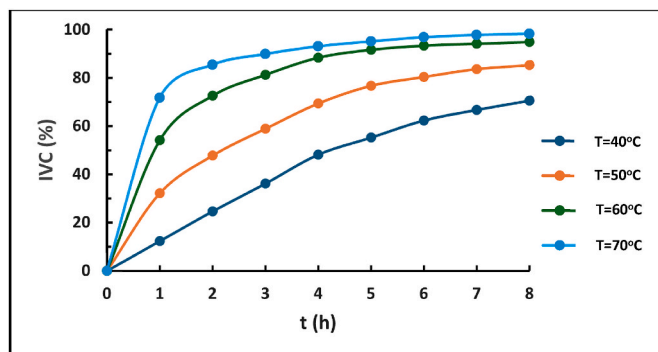
Three potential reactions can occur during the epoxidation of sunflower oil using an acid catalyst [23]:

Reaction 1: Formation of peroxyformic acid.

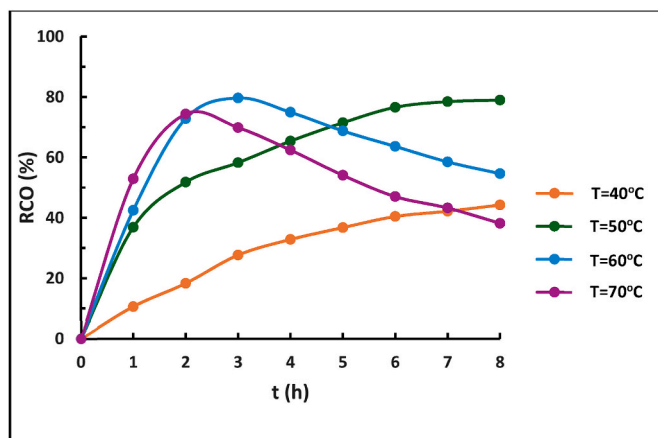
**Table 1**

Composition of fatty acids in sunflower oil.

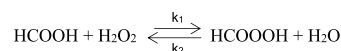
Fatty acid	Composition (%)
Lauric acid	0.02
Myristic acid	0.18
Palmitic acid	5.73
Stearic acid	2.00
Oleic acid	38.55
Linoleic acid	53.52



**Fig. 1.** Effect of temperature and reaction time on iodine conversion.



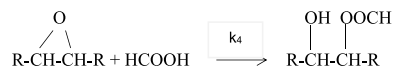
**Fig. 2.** Effect of temperature and reaction time on oxirane conversion.



Reaction 2: Formation of the oxirane ring (primary reaction).



Reaction 3: Formation of hydroxy formate (side reaction).



### 3.2. Effect of molar ratio and reaction time

The influence of the molar ratio of double bonds to hydrogen peroxide at 60 °C on iodine value conversion and conversion to oxirane was examined within the range of 1:1.5 to 1:2.5. An increase in the initial molar ratio of double bonds to hydrogen peroxide significantly affected the iodine value conversion for the epoxidation oil utilizing in situ generated peroxyformic acid (Fig. 3). However, this impact was less pronounced at the molar ratio of 1:2 and 1:2.5, as evidenced by the nearly overlapping plots for these two ratios. The iodine value conversion achieved 82 % with a molar ratio of double bonds to hydrogen peroxide 1:1.5 at 8 hours of reaction time. This value was lower than that of epoxidation at a molar ratio 1:2.5 and attained about 98 % iodine conversion within 8 hours. Therefore, the optimal molar ratio and reaction time for iodine conversion were achieved at 1:2.5 and 8 hours.

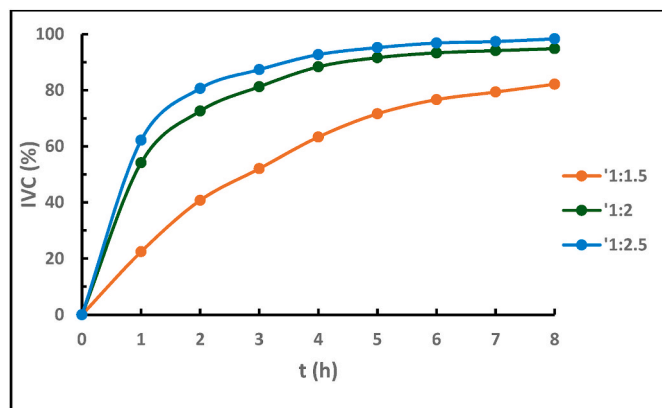


Fig. 3. Effect of molar ratio and reaction time on iodine conversion.

The effect of double bonds to hydrogen peroxide mole ratio on oxirane conversion can be illustrated in Fig. 4. Oxirane conversion initially increased and then decreased as the reaction time peaked. Prolonged reaction time leads to the opening of oxirane rings, potentially forming side products from the epoxidation reaction. The differences in oxirane conversion across the 3 molar ratios are minimal, particularly the maximum relative conversion to oxirane in the 70–80 % range. However, the 1:2 molar ratio of double bonds to hydrogen peroxide seems to be the optimal concentration for the epoxidation of sunflower oil as it attained 80 % conversion to oxirane. Work on the epoxidation of cottonseed oil shows similar results with molar ratio double bonds to hydrogen peroxide 1:2 is the optimum condition for the molar ratio variables [17].

### 3.3. Kinetics of the epoxidation reaction

The epoxidation of sunflower oil utilizes in situ-generated peroxyformic acid, produced from formic acid and hydrogen peroxide in the presence of an acid catalyst. This process occurs in two-step reactions: (i) the formation of peroxyformic acid and (ii) the conversion of double bonds to oxirane rings, facilitated by peroxyformic acid [14]. The first step is considered the rate-determining step, assuming that the concentration of peroxyformic acid remains constant throughout the reaction while neglecting any degradation of the oxirane rings. Consequently, the overall rate equation for the epoxidation reaction can be expressed as:

$$\frac{d[EP]}{dt} = k\{[H_2O_2]_0 - [EP]\}[HCOOH]_0 \quad (4)$$

In this context, the notation "EP" represents epoxides, while the subscript "o" denotes the initial concentrations. The integrating equation (4)

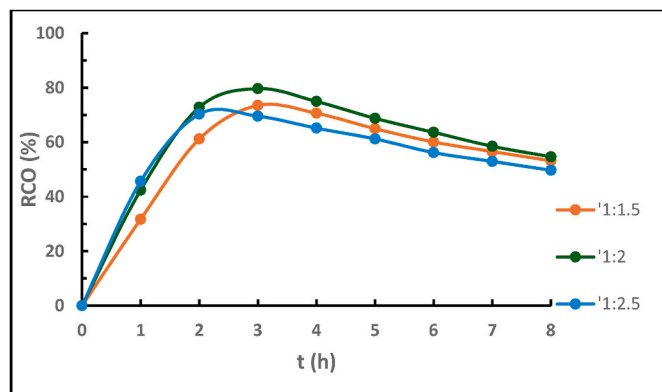


Fig. 4. Effect of molar ratio and reaction time on oxirane conversion.

results in equation (5), which allows the estimate of the reaction rate constant ( $k$ ).

$$\ln\{[H_2O_2]_0 - [EP]\} = -k[HCOOH]_0 t + \ln[H_2O_2]_0 \quad (5)$$

The rate constant ( $k$ ) for each temperature can be obtained from equation (5) as the slope of the plot  $\ln\{[H_2O_2]_0 - [EP]\}$  against reaction time ( $t$ ). The summarized results are presented in Table 2.

From the plot of  $\ln(k)$  against  $1/T$ , the activation energy ( $E_a$ ) was found to be 19.4 kJ/mol (4.66 kcal/mol). This activation energy value is considerably lower than that for epoxidation of mustard oil using peroxyacetic acid generated in situ, which was determined to be 43 kJ/mol, while employing the same kinetics model [16].

### 3.4. FTIR spectra

Fig. 5 presents the FT-IR spectra of epoxidized oil at various reaction times. The peaks observed at  $3009\text{ cm}^{-1}$  and  $825$  or  $843\text{ cm}^{-1}$  correspond to the C=C double bond and the oxirane ring (C–O–C) [24], respectively. The presence of hydroxyl (OH) groups, indicative of glycols, was also detected during the epoxidation reaction, although at low intensity around  $3450\text{ cm}^{-1}$  [15]. These spectra confirm that, as the reaction progresses with reaction time, the double bonds in sunflower oil are converted into oxirane rings, indicating the formation of the oxirane ring (C–O–C). Furthermore, a competing reaction involving the opening of oxirane rings appears to occur, forming substances containing OH groups.

## 4. Conclusions

The kinetics of the epoxidation of sunflower oil were studied, revealing that temperature, reaction time, and the molar ratio of double bonds to hydrogen peroxide significantly influenced the increase in iodine value conversion. At low temperatures, the conversion of double bonds to oxirane rises with increasing reaction temperature and time, reaching a peak before declining at higher temperatures. A higher molar ratio of double bonds to hydrogen peroxide enhances the conversion of iodine value, although it reduces the conversion to oxirane after a particular reaction duration. The optimal conditions for epoxidation identified in this study include a temperature of  $60\text{ }^\circ\text{C}$ , a reaction time of 3 hours, and a 1:2 mol ratio of double bonds to hydrogen peroxide, corresponding to the maximum conversion to oxirane. The reaction constants at  $40\text{ }^\circ\text{C}$ ,  $50\text{ }^\circ\text{C}$ ,  $60\text{ }^\circ\text{C}$ , and  $70\text{ }^\circ\text{C}$  ranged from  $7.7$  to  $14.6 \times 10^{-6}$  liter/mol.s with an activation energy of 19.4 kJ/mol. The conversion of double bonds in sunflower oil into oxirane rings was monitored by observing the functional groups in the epoxidized oil throughout the epoxidation reaction. While this study provides valuable insights into the kinetics of sunflower oil epoxidation using in situ-generated peroxyformic acid, further research is needed to enhance the efficiency and applicability of this process. Future studies should optimize reaction conditions using advanced statistical approaches to maximize conversion to oxirane while minimizing side reactions.

### CRedit authorship contribution statement

**Edy Purwanto:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation,

Table 2

The rate constant of sunflower oil epoxidation.

T (°C)	T (K)	1/T (K <sup>-1</sup> )	k × 10 <sup>6</sup> (l/mol.s)
40	313.15	0.0031934	7.7
50	323.15	0.0030945	10.9
60	333.15	0.0030017	13.6
70	343.15	0.0029142	14.6



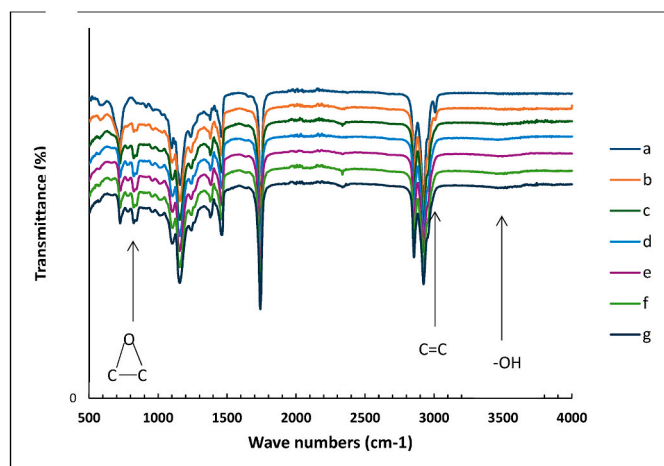


Fig. 5. FT-IR spectra of epoxidized oil for different reaction time (a) 0 hour (b) 1 hour, (c) 2 hours, (d) 3 hours, (e) 4 hours, (f) 5 hours (g) 8 hours.

Conceptualization. **Jericho Stevanno Budianto:** Methodology. **Matthew Marcelino Indrawanto:** Resources, Methodology. **Aloisiyus Yuli Widiyanto:** Investigation, Conceptualization. **Puguh Setyoprato:** Investigation, Conceptualization.

#### Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Edy Purwanto, Puguh Setyoprato, Aloisiyus Yuli Widiyanto reports financial support was provided by Kementerian Pendidikan, Kebudayaan, Riset, dan Teknologi, Indonesia. - If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Data availability

Data will be made available on request.

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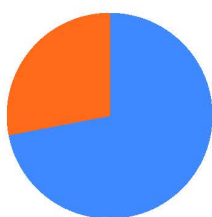
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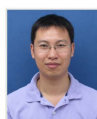
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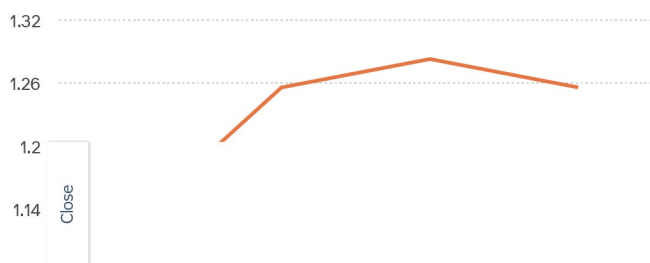
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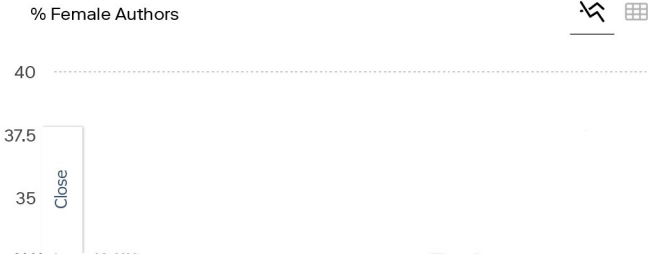
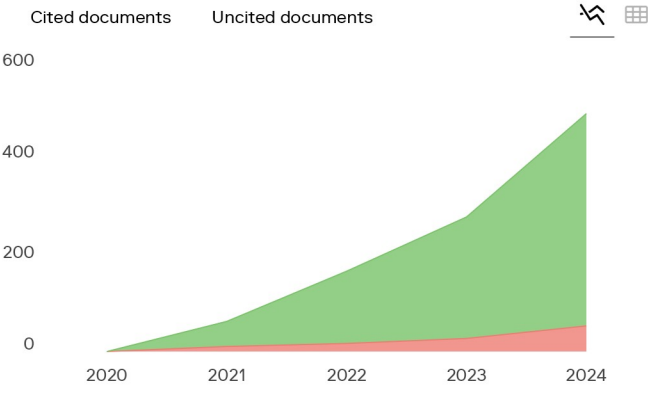
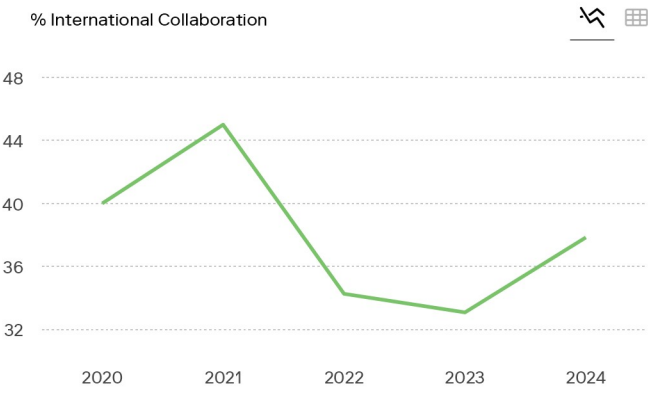
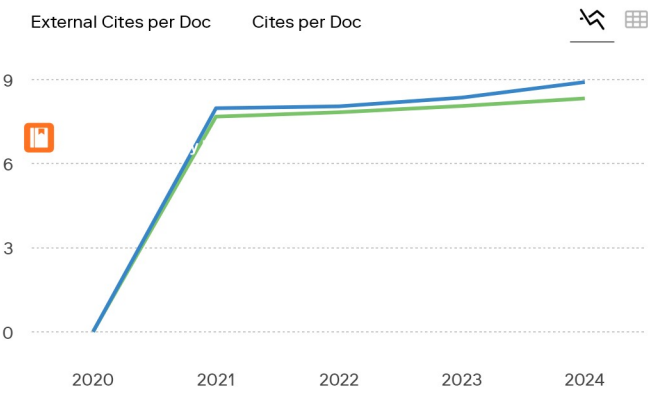
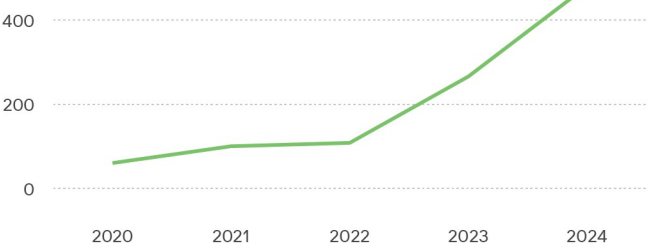
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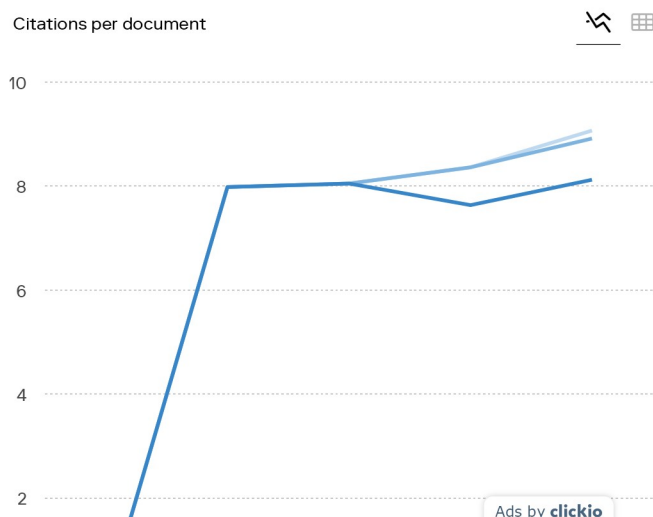
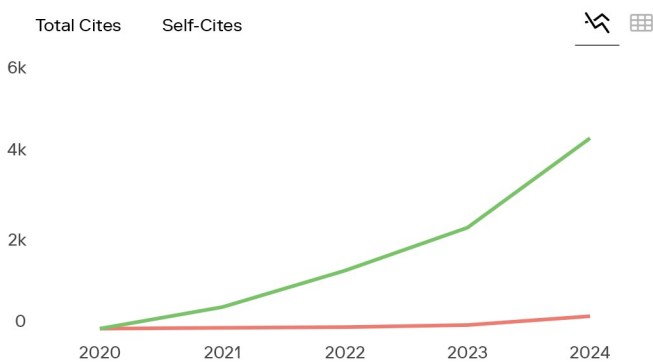
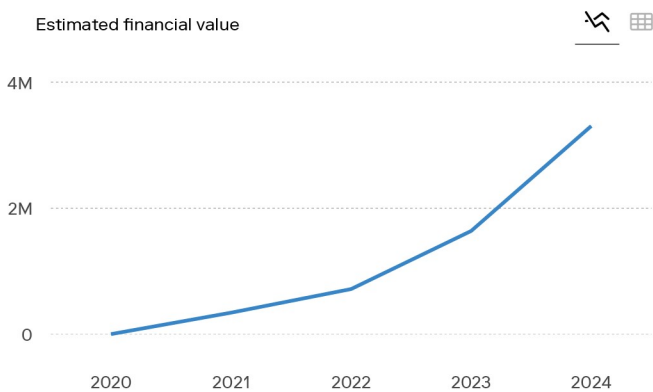
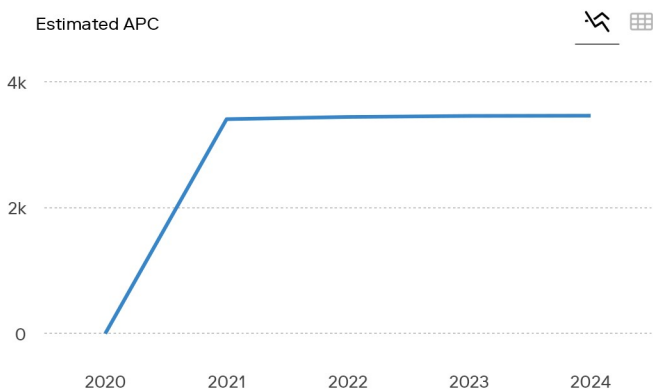
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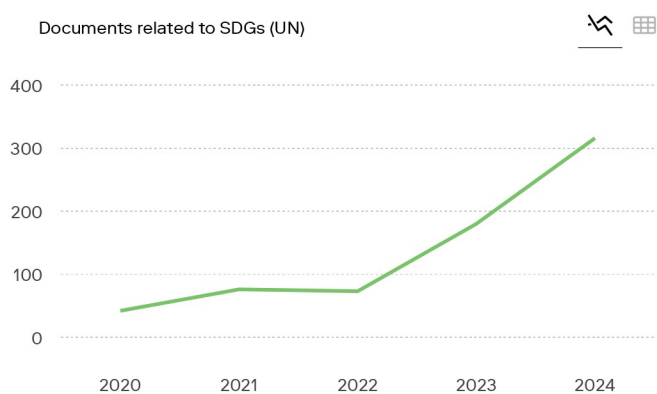
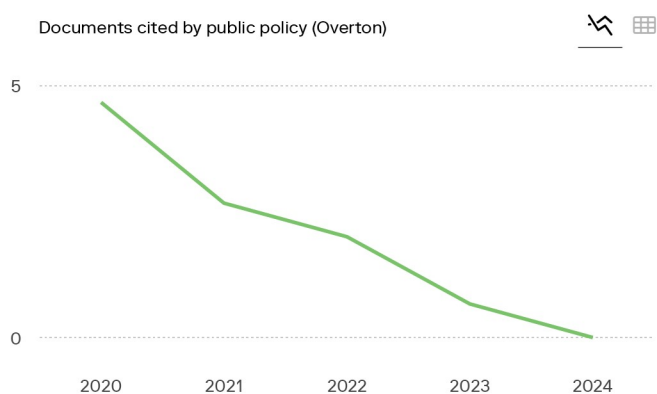
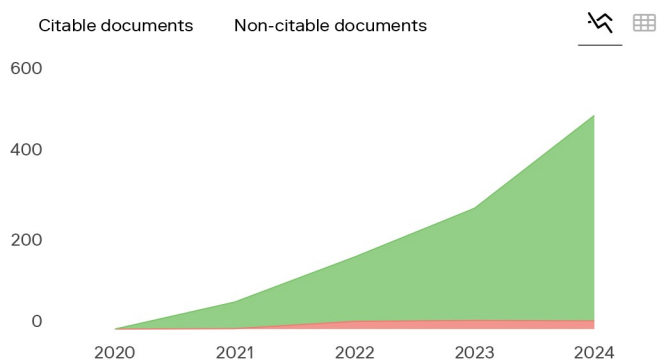
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