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# Rice Husk Ash for the Stabilization of the Outer Interfacial Layer of W/O/W Double Emulsion

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**Abstract.** Many food emulsions based on oil-in-water (O/W) emulsion such as cream, mayonnaise, dressings, and so on were quite important to improve the dish savory. Recently, low-fat products gained some attraction due to the increasing awareness of the people regarding healthy food and healthy lifestyle. The development of water-in-oil-in-water (W/O/W) emulsion seemed quite promising in order to reduce the oil content of the whole emulsion. However, the stability of W/O/W emulsion was hardly maintained as compared to those of O/W single emulsion. Rice husk silica which tended to be hydrophilic was used to stabilize the outer interfacial layer with some pH adjustment. The inner emulsion was prepared by dispersing the palm oil into inner aqueous phase containing the mixture of Tween 20 and Span 80. The inner emulsion was then dispersed into the outer aqueous phase containing rice husk ash of which pH was previously adjusted (2, 3, and 4). The emulsification was conducted using a rotor stator homogenizer. The results showed that the highest W/O/W stability was achieved when silica was dispersed at very low pH of 2. The decay rate obtained from exponential decay model fitted into the destabilization curve of emulsion prepared at pH 2 was much lower with the order of 2 as compared to those obtained from emulsions prepared at higher pH's. The emulsion stability maintained at 80% after 7 days and estimated half time of about 0.92 day. The pickering double emulsion with rice husk silica seemed promising to substitute the use of polymeric emulsifiers besides providing a more stable emulsions against heat and shear during the food processing.

**Keywords:** double emulsion, exponential decay, pH, rice husk ash, W/O/W

## INTRODUCTION

Emulsion is a heterogenous system which is frequently found in processed foods. Mayonnaise which is known as a creamy and viscous condiment mainly used for the preparation of salads, dips, and burgers is an example of oil in water emulsion. A relatively high oil content of about 65-75% [1] is dispersed in aqueous phase of mayonnaise imparting to its viscous characteristics. The high fat food products have become the main big issue in food industries since the reduction of fat may lead to a reduced palatability or creaminess. The use of water-in-oil-in-water (W/O/W) could become an alternative for the development of low-fat mayonnaise without affecting its physical properties [2]. The additional dispersion of internal aqueous phase inside the oil droplets could reduce the overall fat concentration in the mayonnaise product. However, the stability of the multiple emulsion has become the other challenge to solve since two interfacial layers are existing in the system.

Emulsion is a thermodynamically unstable system; however, the kinetic stability could be improved by adding emulsifiers to reduce the interfacial tension. The use of particle instead of polymeric emulsifiers to stabilize the emulsion which is so called Pickering emulsion has been introduced in 1907 [3]. Nevertheless, the polymeric emulsifiers have become more rapidly developed and massively used in the food industries due to its high efficiency. Recently, the interest of developing Pickering emulsion has been increasing since it is highly stable against pH and temperature changes, coalescences, Oswald ripening, and lipid oxidation [4-6]. Pickering emulsion

has been increasingly applied in several fields, such as food, pharmaceuticals, and cosmetics [7, 8]. Many particles such as gelatin, cellulose, graphene, silica, etc have been used in the formation of pickering emulsions [5, 9-11].

Biosilica particles have been widely used in developing pickering emulsions, especially for stabilizing oil in water emulsion [12-15]. Biosilica is referred to silica, which is accumulated in living tissues, which has an amorphous form and tends to be hydrophilic due to the presence of hydroxyl group in the form of silanol Si-OH on its surface [13]. Rice husk silica has become one of the biosilica resources derived from plants. According to U.S. Food & Drug Administration [16], rice husk silica is safe and could be used as food additive. Besides that, rice husk silica has no toxicity and harmless in contrast to crystalline silica [17, 18].

The effectiveness of using particles to stabilize O/W emulsion were dependent on particle size and shape, inter-particle interactions and the wettability of particles in both phases [19]. Binks and Lumsdon [19] and Pichot *et al.* [20] used colloidal silica to stabilize O/W emulsion. Biosilica derived from rice husks combined with polymeric emulsifiers such as lecithin and Tween-20 has been used for the formation of O/W pickering emulsions [12-14]. The addition of salt and pH adjustment has been carried out to modify the wettability thus its hydrophilicity characteristic of silica particle [21, 22]. Liu *et al.* [23] investigated the effect of pH on the stability of toluene in water emulsion. Nanosilica particles were combined with zwitterionic surfactant. The results showed that the emulsion was stable against coalescence at pH below 5 in several months regardless the increase of oil globule with time. Furthermore, the emulsion was totally not stable for the pH above 8.5. The application of rice husk silica for stabilizing W/O/W emulsion in the presence of Tween-20 was also dependent on pH [15].

The objective of this research is to investigate the use of biosilica in the form of rice husk ash (RHA) without the addition of any polymeric emulsifiers in order to stabilize the outer interfacial layer of W/O/W double emulsion with the variation of pH of the outer aqueous phase. As the use of rice husk silica seems promising in producing W/O/W with relatively high stability, the use of synthetic polymeric surfactants as well as the fat fraction incorporated in the processed foods could be far reduced. Furthermore, the internal aqueous phase could also function as encapsulation vehicle of nutrients such as vitamin, mineral, or antioxidants. This would trigger the technology breakthrough towards a more healthful and nutritious food innovation products.

## METHODOLOGY

### Materials

Palm cooking oil (Sunco, PT. Milkie Oleo Nabati Industri, Bekasi, Indonesia); sorbitan monooleate/ Span-80 (Sigma Aldrich, Germany); polyethylene glycol sorbitan monolaurate/ Tween-20 (Merck, Germany); citric acid powder (Merck, Germany); sodium dihydrogen citrate/ monosodium citrate powder (Merck, Germany); disodium hydrogen citrate/ disodium citrate (Merck, Germany); demineralized water. Rice husk was obtained from the paddy milling in Mojokerto, East Java, Indonesia.

### Preparation of rice husk ash

Rice husks were separated from the impurities and then subjected to washing with water and rinsing with demineralized water. Clean rice husks were dried at 105°C for 2 hours in an oven (Mettler, Germany) and then calcined at 750°C for 5 hours in the furnace (Ney VULCAN D-550, Dentsply Ceramco, USA) for the organic compounds removal. The remaining rice husk ash (RHA) which consisted of silica of > 90% was gray in color. The rice husk ash was milled and screened with 200 mesh screens prior to use as the emulsion stabilizer.

### Preparation of W/O emulsion

The aqueous phase containing water added by 2.1% Tween-20 was stirred with a magnetic bar at 300 rpm for 7 minutes until homogenous. Furthermore, the oil phase containing 5.3% Span-80 was mixed with a magnetic bar at 800 rpm for 7 minutes. Afterwards, the aqueous phase with fraction of 40% was dispersed in the oil phase using a rotor stator (IKA T25 digital ULTRA TURRAX, Germany) at mixing speed of 20,000 rpm for 6 minutes. This primary emulsion was highly stable for several weeks.

## Preparation of buffer solution

Outer aqueous phase of W/O/W emulsion with various pH of 2, 3, and 4 was prepared using the buffer solution. The buffer solution of pH 2 was prepared by mixing 47 ml of 0.065 M citric acid with 3 ml of  $4.628 \times 10^{-3}$  M monosodium citrate into the 100 ml volumetric flask. Water was added up to 100 ml and the solution was well shaken. The buffer solution of pH 3 was prepared by mixing 35 ml of 0.0105 M citric acid with 15 ml of  $7.4596 \times 10^{-3}$  M monosodium citrate, while the buffer solution of pH 4 was prepared by mixing 45 ml of  $5.0798 \times 10^{-3}$  M disodium citrate with 15 ml of  $7.4596 \times 10^{-3}$  M monosodium citrate in the 100 ml volumetric flask. The flask was filled with water until reaching the mark and was shaken for several times until homogeneous. The control was prepared without any pH adjustment of outer aqueous phase with pH value of about 5.7.

## Preparation of W/O/W double emulsion

The outer aqueous phase with adjusted pH (2, 3, and 4) was prepared prior to dispersion of primary emulsion to obtain the double emulsion. Rice husk silica particles of 0.5% without any other emulsifier were added into the outer aqueous phase to help stabilize the interfacial layer between oil droplet with the outer aqueous phase. The mixture was mixed using a magnetic stirrer at 800 rpm for 10 minutes.

W/O/W double emulsion was prepared by dispersing the primary emulsion W/O emulsion with the fraction of 0.2 into the outer aqueous phase using a rotor stator (IKA T25 digital ULTRA TURRAX, Germany) at 8000 rpm for 3 minutes. The double emulsion was poured into a transparent 40 ml glass vial (ID= 25 mm, height= 95 mm) and stored at a room temperature of  $\sim 28^\circ\text{C}$ . The percentages used belonged to weight %.

## Determination of W/O/W emulsion stability

The emulsion stability was determined using equation 1. The height of emulsion was measured right after the double emulsion preparation and several consecutive days up to 7 days. The stable emulsion layer was indicated by a milky appearance excluding the oil, cream, and sediment layers.

$$\%S = \frac{h_t}{h_0} \times 100\% \quad (1)$$

whereas  $h_t$  is emulsion height at a certain time and  $h_0$  is initial emulsion height.

## Determination of models of destabilization of W/O/W emulsion

The whole data of emulsion stability up to 7 days was used for the models fitting. The destabilization of O/W emulsion was fitted with the exponential decay model of 2 parameters and 3 parameters using SigmaPlot version 11.0 as shown in Eq. 2 and Eq. 3, respectively. The coefficient of determination ( $R^2$ ) was also determined.

$$S = ae^{-kt} \quad (2)$$

$$S = S_0 + ae^{-kt} \quad (3)$$

whereas  $S$  is the percentage of emulsion stability after time  $t$  and  $S_0$  is the initial emulsion stability percentage,  $a$  is the parameter,  $k$  is the emulsion destabilization rate constant (per day), and  $t$  is the storage time (day).

Furthermore, half time,  $t_{1/2}$  (day) indicated the time required to reach the 50% emulsion stability according to Dyab [24]. The higher the half time is, the more stable the emulsion is. Half time is calculated from the rearrangement of Equation 3 by setting  $S = \frac{1}{2} S_0$  and the calculation is shown by Eq. 4.

$$t_{1/2} = \frac{\ln\left(\frac{a}{\frac{1}{2}S_0}\right)}{k} \quad (4)$$

## RESULTS AND DISCUSSION

The stability of double emulsion W/O/W stabilized by rice husk ash on the outer aqueous phase was significantly influenced by pH. The stability of the double emulsion was the highest on pH 2 and was decreasing as the pH was increasing up to pH 5.7 as seen in Fig. 1. After 7 days, the stability of the W/O/W emulsion prepared at pH 2 was maintained at 80%, whereas the stability other samples prepared at pH above 2 dropped to between 50 and 70%. Furthermore, the stability of control sample which was prepared without RHA was quite instable with the stability of about 25% after 7-day storage as depicted in Fig. 2. These results implied that biosilica in the form of RHA had the ability to stabilize the outer aqueous phase by adhering on the interfacial layer between oil droplets and outer aqueous phase.

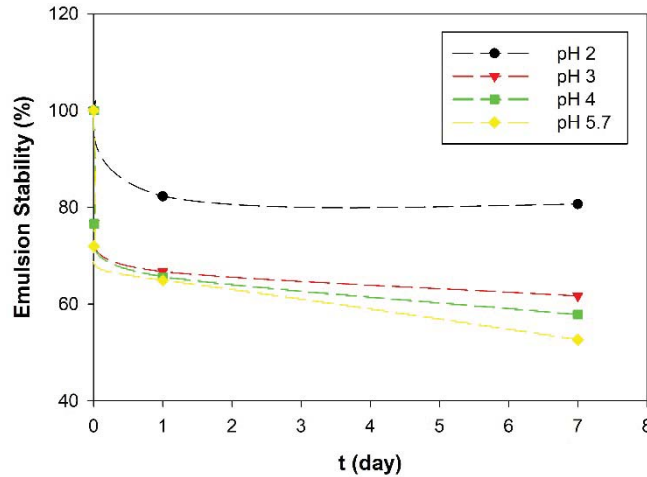


FIGURE 1. Stability of W/O/W double emulsion prepared with 0.5% RHA at various pH of the outer aqueous phase

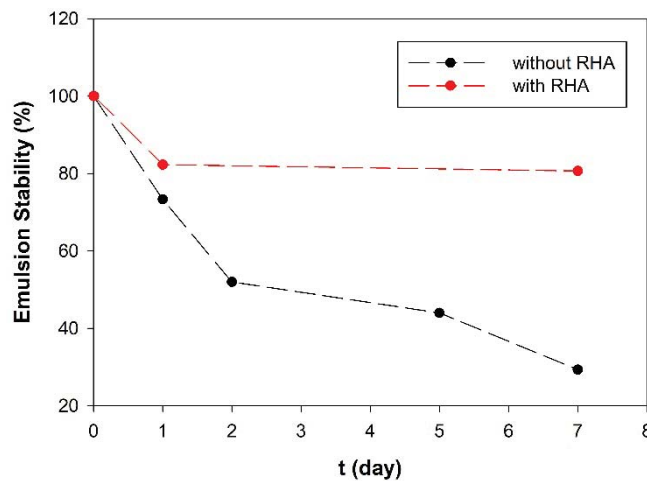
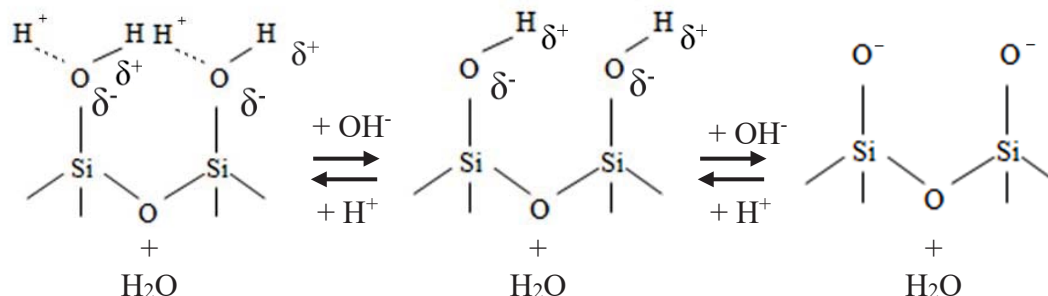


FIGURE 2. Stability of W/O/W double emulsion prepared with 0.5% RHA and without RHA at pH 2

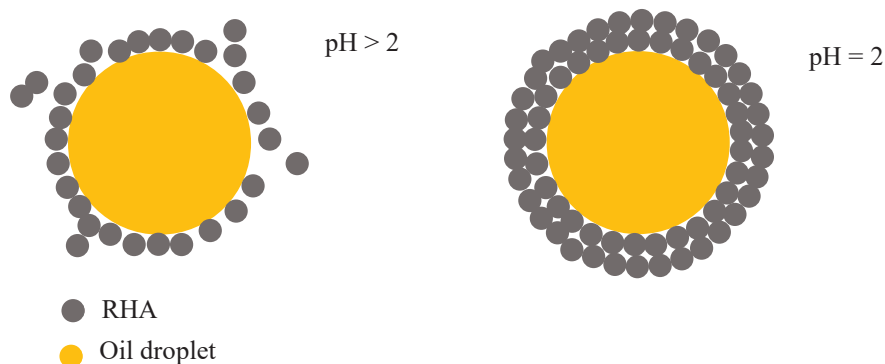
Many studies demonstrated the dependence of emulsion stability stabilized by silica on the pH value [15, 20]. Pichot *et al.* [20] investigated the stability of O/W emulsion using bare silica particles at pH 2 till 10. He observed that the use of biosilica was more effective in stabilizing the emulsion when pH was decreasing. When the pH system was approaching acidic ( $\text{pH} < 4$ ), the biosilica particle was tended to be chargeless thus favoring the formation of closely packed particles aggregation surrounding the interfacial layer between oil droplets and water. In turn, this barrier prevented the oil flocculation and coalescences thus improving the overall emulsion stability. On

the other hand, the increasing pH value resulted in a decreasing stability of the emulsion. As the pH was approaching the basic region, biosilica particles tended to be dissociated to form a more hydrophilic  $\text{SiO}^-$  group [24]. These negative charges on the biosilica surfaces would in turn induce the repelling forces between biosilica particles inhibiting the formation of solid biosilica aggregates surrounding the interfaces of oil and water [15]. The reaction mechanisms of biosilica dissociation dependent on pH could be seen in Fig. 3.



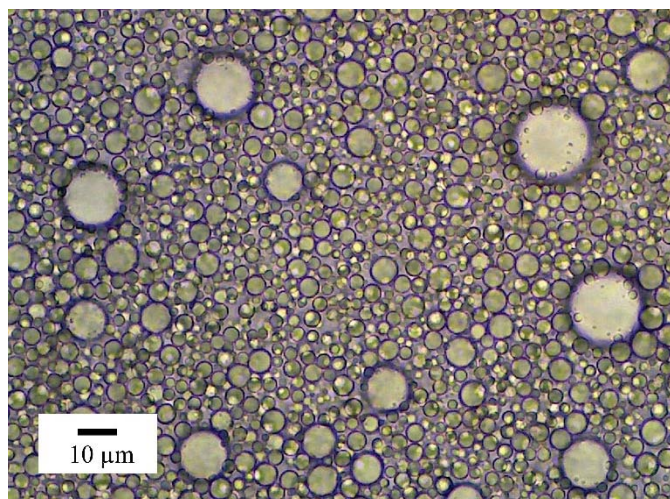
**FIGURE 3.** The reaction mechanisms of biosilica dissociation dependent on pH

It was obvious that the stability of W/O/W double emulsion stabilized by RHA was the most stable at pH 2 compared to those prepared at higher pH values. Biosilica dissociation did not occur at pH 2, which is also the isoelectric point of natural silica particles [24]. At this point, silica particles possess zero charge, thus fostering aggregation amongst silica particles. The aggregation was likely to be closely packed as could be influenced by the strong hydrogen bonding interactions among the hydroxyl groups on the biosilica surfaces. The contact angle between silica particle and water could also affect the wettability of the particle and thus their position in the interfacial layers [25]. When the pH of outer aqueous phase was less than 2 or more than 2, the biosilica particles would become charged due to the formation of  $\text{Si-OH}_2^+$  and  $\text{SiO}^-$ , respectively. These either positively or negatively charged biosilica particles would repel one another therefore preventing the formation of the multiple layers of biosilica aggregates on the interfacial layers between oil and water. Furthermore, the negatively charged biosilica formed at pH above 2 would tend to be suspended on the continuous phase rather than adsorbing on the oil-water interfaces. The illustration of biosilica aggregates formation at pH 2 and at pH higher than 2 was depicted in Fig. 4. This mechanism clearly explained the remarkable stability of the double emulsion prepared at pH 2 in contrast to those prepared on higher pH's as depicted in Fig. 1.



**FIGURE 4.** The illustration of biosilica aggregates formation at pH 2 and at pH higher than 2

The microstructure of W/O/W double emulsion stabilized by 0.5% RHA on the outer aqueous phase prepared at pH 2 was depicted in Fig. 5. Even though the overall double emulsion stability was retained at 80% after 7-day storage, the presence of internal water droplets inside the oil droplets was very few. This probably caused by the differences in ionic concentration between the inner aqueous phase and outer aqueous phase. The outer aqueous phase contained some salts as they were added to act as buffering solution whereas the internal aqueous phase was only demineralized water. It was plausible that the internal water droplet migrated to the outer aqueous phase via the oil droplet with time. This may also be affected by the instability of the internal aqueous phase due to many factors such as flocculation or coalescence and the attachment of the emulsifiers between internal aqueous phase and oil droplets.

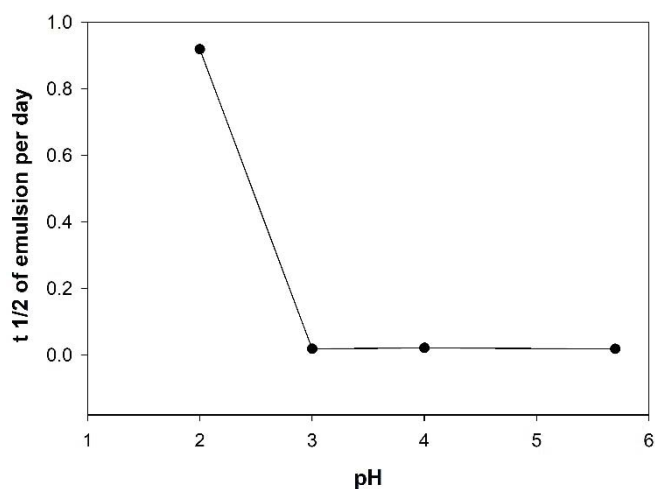


**FIGURE 5.** The microstructure of W/O/W double emulsion stabilized by 0.5% RHA on the outer aqueous phase prepared at pH 2.

The decrease of W/O/W double emulsion stability with time during 7-day storage was modelled using the exponential decay models of 2 and 3 parameters. The values of destabilization rate constants,  $k$  derived of those models could be seen in Table 1. It was obvious that coefficient of determination ( $R^2$ ) values derived from the fitting of the data with the model of 3 parameters approaching to 1 in contrast to those obtained using the model of 2 parameters. This inferred the best fit of the exponential decay of 3 parameters to the experimental data. Furthermore, the lowest  $k$  of about  $2.8 \text{ day}^{-1}$  was obtained for the W/O/W double emulsion stabilized by 0.5% biosilica prepared at pH 2. The  $k$  values of other samples prepared at higher pH were much higher with the magnitude order of about 2. Moreover, the calculated half time ( $t_{1/2}$ ) of the destabilization process of W/O/W double emulsion with time could be seen in Fig. 6. Again, the highest stability of W/O/W double emulsions stabilized by RHA prepared at pH 2 was pronounced indicating by its highest half time of about 0.92 day or about 22 hours. The double emulsion prepared at higher pH's showed half time of approaching 0 day or about half an hour indicating the instantaneous destabilization process of the emulsion. These whole results again corroborated the importance of acidic pH as low as 2 for the attainment of the highest stability of W/O/W double emulsions when biosilica in the form of RHA was used to stabilize the interfacial layers between oil droplet and the outer aqueous phase.

**TABLE 1.** Destabilization rate constants and  $R^2$  values according to exponential decay models of 2 and 3 parameters for W/O/W double emulsions stabilized by 0.5% RHA at various pH of the outer aqueous phase stored consecutively up to 7 days.

| pH  | Exponential decay, 2 parameters |        | Exponential decay, 3 parameters |        |
|-----|---------------------------------|--------|---------------------------------|--------|
|     | $k \text{ (day}^{-1}\text{)}$   | $R^2$  | $k \text{ (day}^{-1}\text{)}$   | $R^2$  |
| 2   | 0.026                           | 0.5382 | 2.8                             | 0.9998 |
| 3   | 0.044                           | 0.4762 | 152.6                           | 0.9856 |
| 4   | 0.053                           | 0.5613 | 137.3                           | 0.9698 |
| 5.7 | 0.062                           | 0.6146 | 165.5                           | 0.9376 |



**FIGURE 6.** Half time ( $t_{1/2}$ ) of the destabilization process of W/O/W double emulsion stabilized by 0.5% RHA prepared at various pH of the outer aqueous phase.

## CONCLUSION

W/O/W double emulsion demonstrated a higher stability when rice husk silica in the form of RHA was used to stabilize the outer interfacial layer. However, pH of the outer aqueous phase played a significant role in modifying the surface charge of biosilica particles thus dictating their particulate aggregation on the interfacial layer. At pH 2, the biosilica particles tended to be chargeless thus the particles were supposed to be aggregated in a densely packed manner. It could form a multilayer serving as a good barrier against flocculation and coalescences imparting to its high stability in contrast to those prepared at pH higher than 2. These results were corroborated by the lowest destabilization rate constant and the highest half time of about  $2.8 \text{ day}^{-1}$  and 0.92 day, respectively obtained for the double emulsion prepared at pH 2 estimated using the exponential decay 3-parameters model. This indicated the highest emulsion stability which still retained at about 80% after 7-day storage. However, only few droplets of internal aqueous phase were seen inside the oil globules probably due to ionic concentration differences between the internal and external aqueous phase. Several parameters are still needed to be optimized and other ingredients might be added in order to improve the stability of the overall W/O/W double emulsion. RHA seems promising to stabilize the outer interfacial layer for developing low food calorie emulsion-based products.

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Elieser Tarigan

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Marta Devega Yuharma, W. Widiyastuti, Ni Made Intan Putri Suari and Heru Setyawan

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Emma Savitri, Maria Irine Tjahayani and Anastasia Apriyani Ngene Say

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**Hate speech content  
detection system on Twitter  
using K-nearest neighbor  
method**

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Vincentius Riandaru Prasetyo and Anton Hendrik  
Samudra

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**School finder, intelligent  
recommendation system for  
elementary school selection**

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Susana Limanto, Endah Asmawati and Yusuf Wira  
Kencana Putra

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system based on the  
application of decision tree  
algorithm in data mining with  
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topic of undergraduate's  
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Yenny Sari, Vincentius Riandaru Prasetyo and Kevin  
Liyansah

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using convolutional neural**

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**Implementing directed pairwise judgement approach in web-based AHP survey application to reduce inconsistency ratio**

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Daniel Hary Prasetyo

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Joko Siswanto, Ida Bagus Made Artadana and  
Muhammad Z. F. N. Siswanto

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Alexander Yohan, Nai-Wei Lo and Kevin Valentino

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Delta Ardy Prima





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Andre and Marcellinus Ferdinand Suciadi

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## Genetic algorithm with adaptive diversification and intensification for the vehicle routing problem

Eric Wibisono, Iris Martin and Dina Natalia Prayogo


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AIP Conference Proceedings **2470**, 050012 (2022);  
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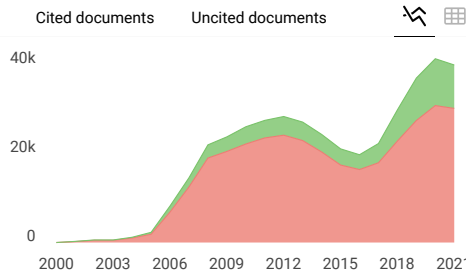
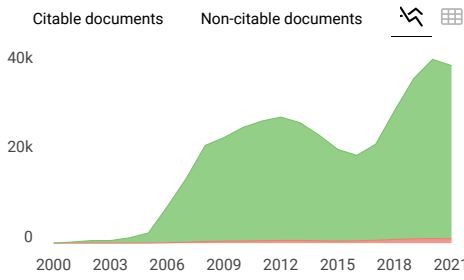
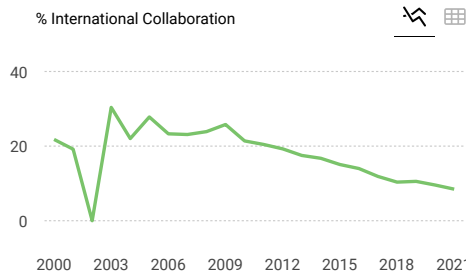
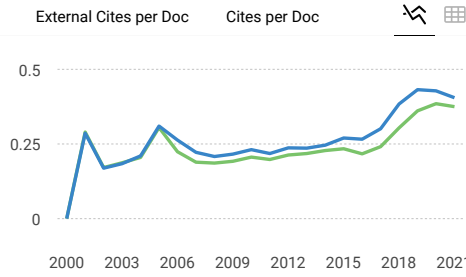
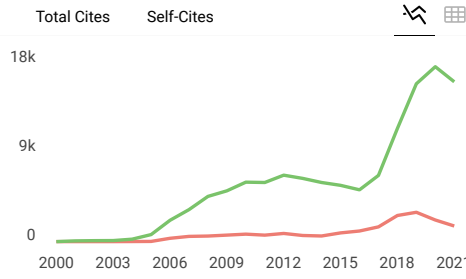
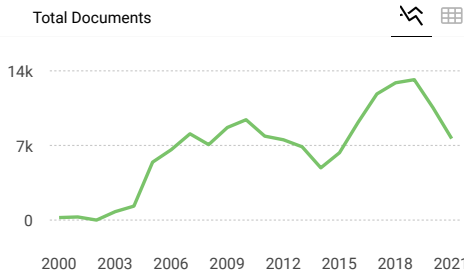
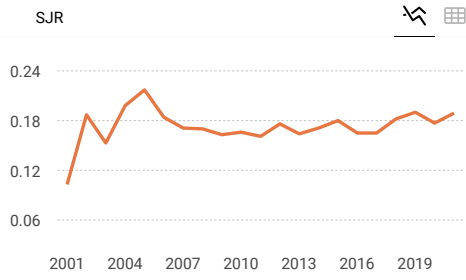
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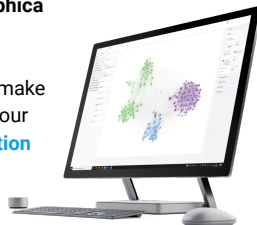
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