Microwave-assisted green synthesis of ZnO nanoparticles using roselle calyx extract: Surface properties and photocatalytic performance

Thanaporn Chueamuang[1], Thanatcha Sonklin[1], Mai Takashima[2], Bunsho Ohtani[3], and Pradudnet Ketwong[1*]

[1]Division of Chemistry, Department of Physical and Material Sciences, Faculty of Liberal Arts and Science, Kasetsart University, Kamphaeng Saen Campus, Nakhon Pathom 73140, Thailand
[2]School of Engineering, Tokyo University of Science, Tokyo 125-8585, Japan
[3]Nonprofit Organization touche NPO, Sapporo 060-0004, Japan
*Corresponding author's e-mail address: pradudnet.k@ku.th

Introduction

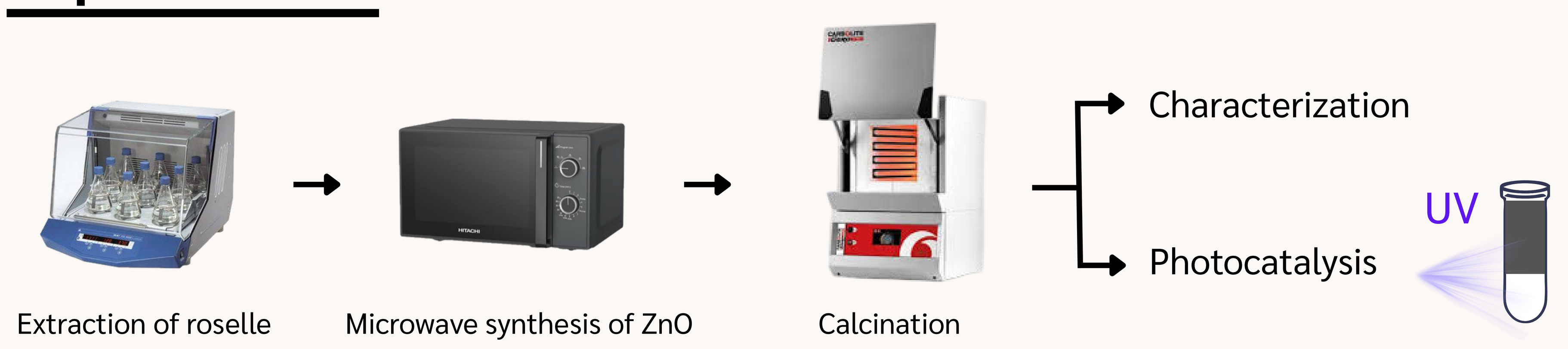
Zinc oxide nanoparticles (**ZnO-NPs**) are widely recognized as effective photocatalysts for environmental remediation owing to their high chemical stability and strong photoactivity. However, conventional synthesis methods often involve toxic reagents and high energy consumption. Green synthesis using plant extracts has emerged as a sustainable alternative, where phytochemicals, particularly flavonoids, play a crucial role in controlling nanoparticle nucleation, growth, and surface properties.

In this study, flavonoid-rich **Kamphaeng Saen roselle** calyx extract was employed as a green reagent for the rapid microwave-assisted synthesis of ZnO-NPs. The synthesized ZnO-NPs exhibited a highly crystalline hexagonal structure with irregular particle shape. Surface characteristics and photocatalytic performance were systematically investigated, with particular emphasis on surface electron trap density. The results highlight the dominant role of surface properties over bulk structure in governing photocatalytic activity and reveal the potential of green-synthesized ZnO nanoparticles as efficient and sustainable photocatalysts for environmental remediation.

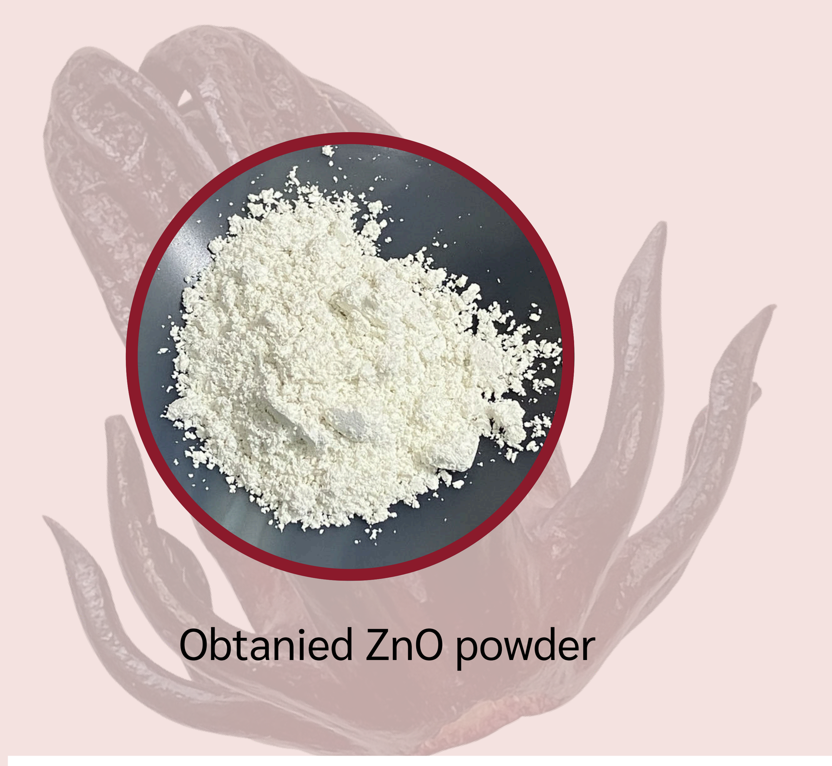
Objectives

1. To synthesize ZnO NPs using roselle calyx extract through microwave-assisted.
2. To investigate the relationship between the bulk and surface properties of synthesized ZnO-NPs and their photocatalytic performance toward formaldehyde (HCHO) degradation.

Experimental



Results



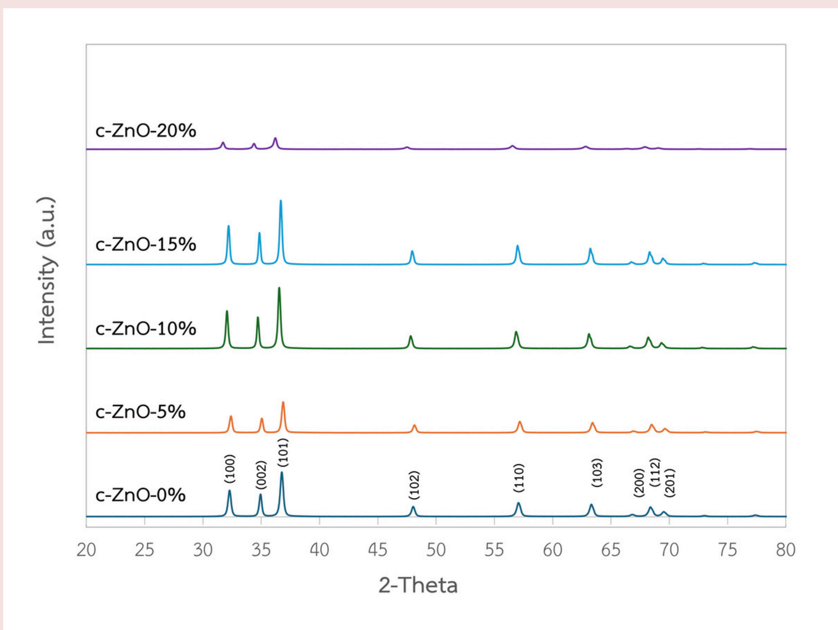


Figure 1. XRD patterns of ZnO NPs.

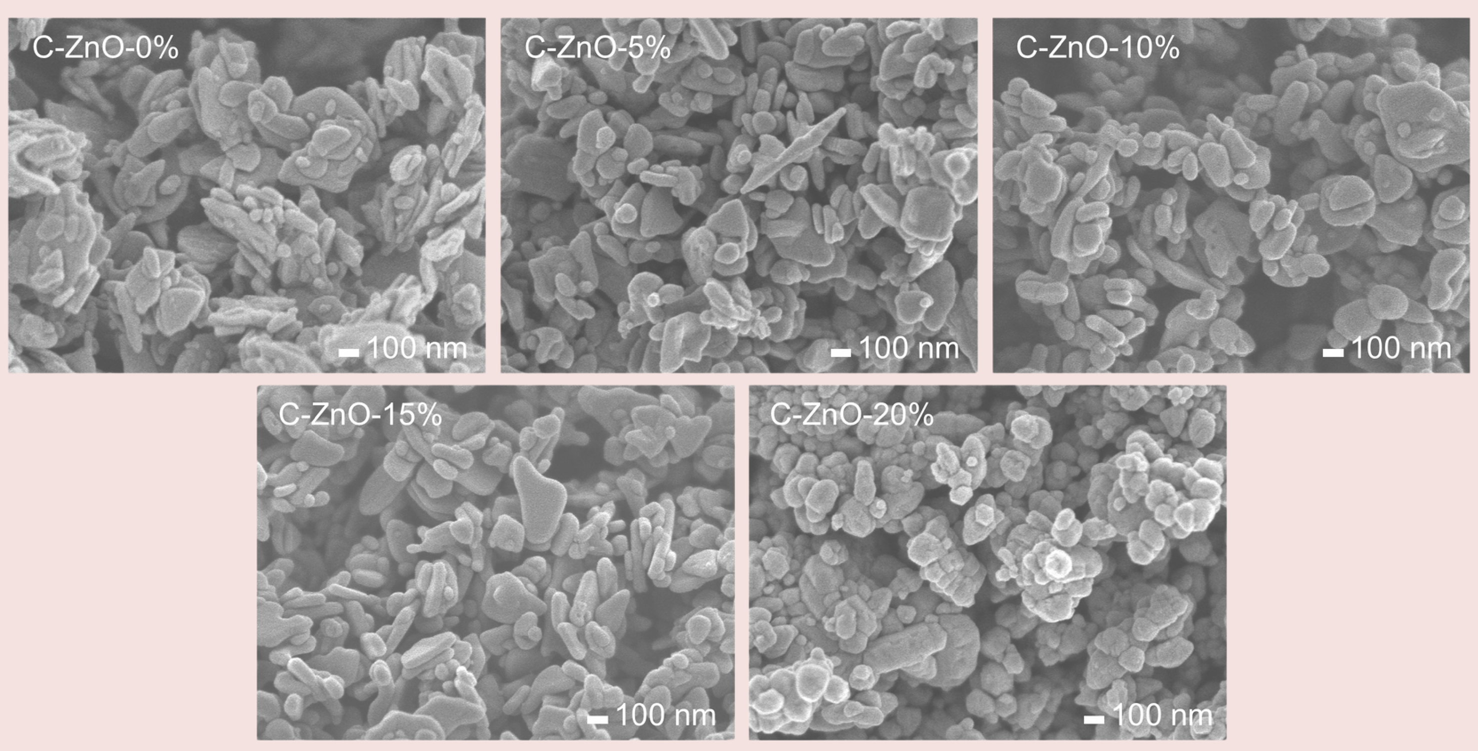


Figure 2. FE-SEM images of ZnO NPs.

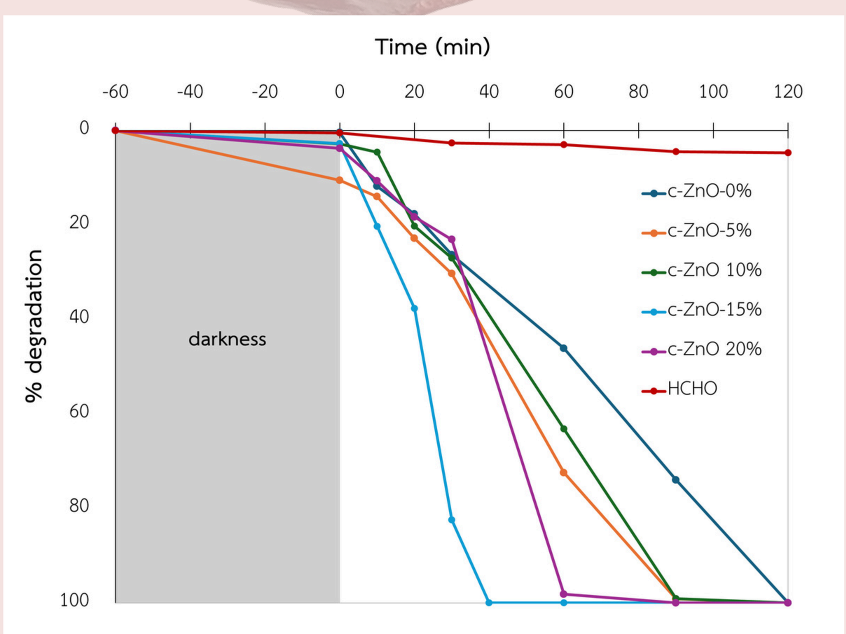


Figure 3. The percentage degradation of HCHO over ZnO NPs by times under UV light irradiation.

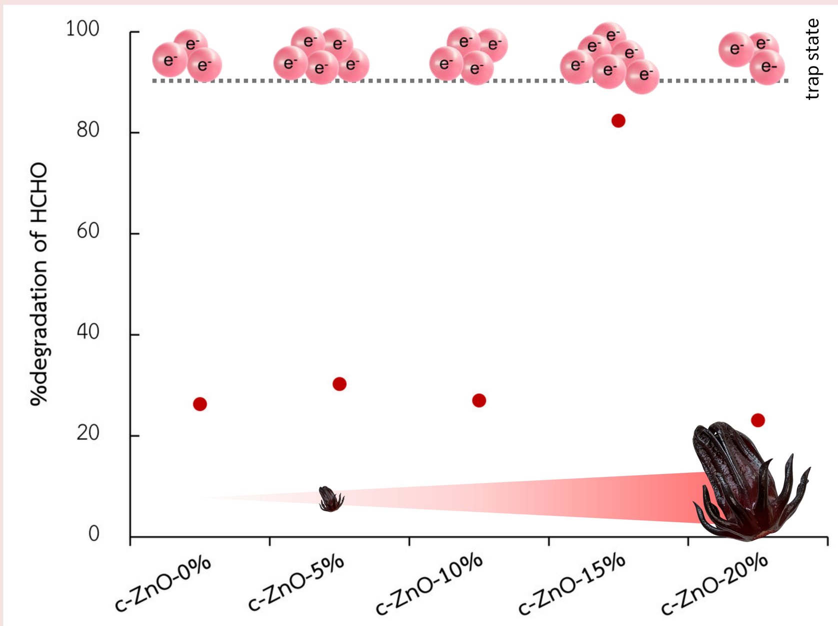


Figure 4. Comparison of photocatalytic activities of ZnO NPs with roselle extract concentration and electron trap densities.

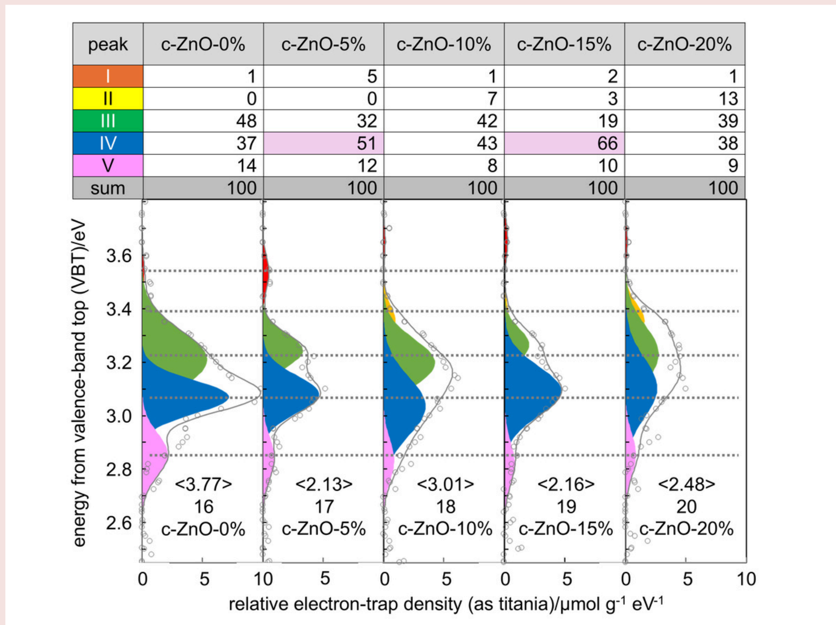


Figure 5. The energy-resolved distribution of the electron trap pattern of ZnO-NPs samples with a table showing the %composition of five peaks.

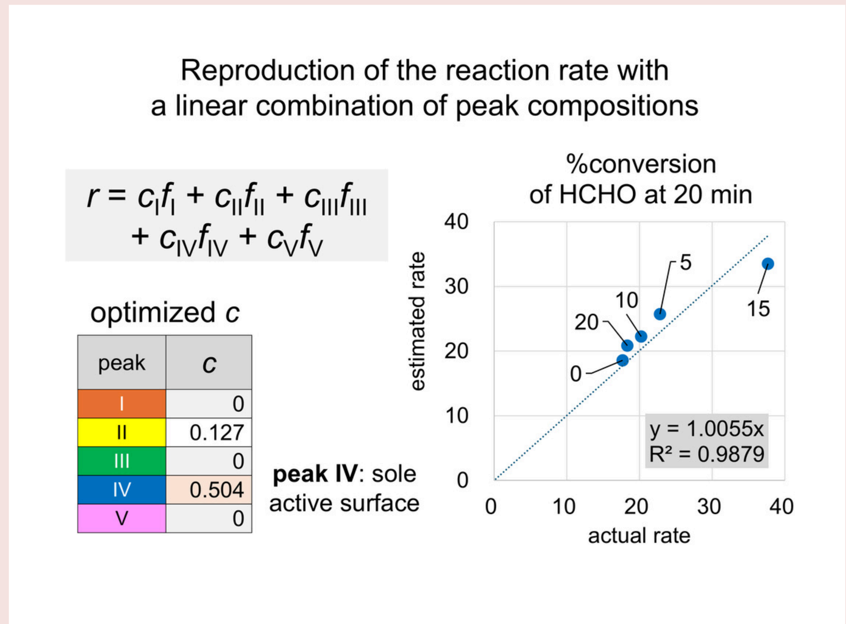


Figure 6. Statistical analysis of rate of HCHO conversion .

Table 1 Characterization and photocatalytic activities of ZnO-NPs samples.

Sample	Crystallite size (nm)	Eg (eV)	SSA (m²/g)	Morphology	Particles size (nm)	%degradation*
c-ZnO-0%	23.93	3.24	9.78	irregular	50-180	26.3
c-ZnO-5%	24.00	3.25	7.77		50-140	30.29
c-ZnO-10%	25.83	3.25	7.97		50-120	27.03
c-ZnO-15%	31.09	3.26	7.56		50-150	82.43
c-ZnO-20%	21.16	3.23	7.39		120	23.06

* Photodegradation of HCHO was performed under UV light (365 nm) using 0.5 mM HCHO solution (30 mL) with 30 mg of photocatalyst. The degradation efficiency was determined at 30 min.

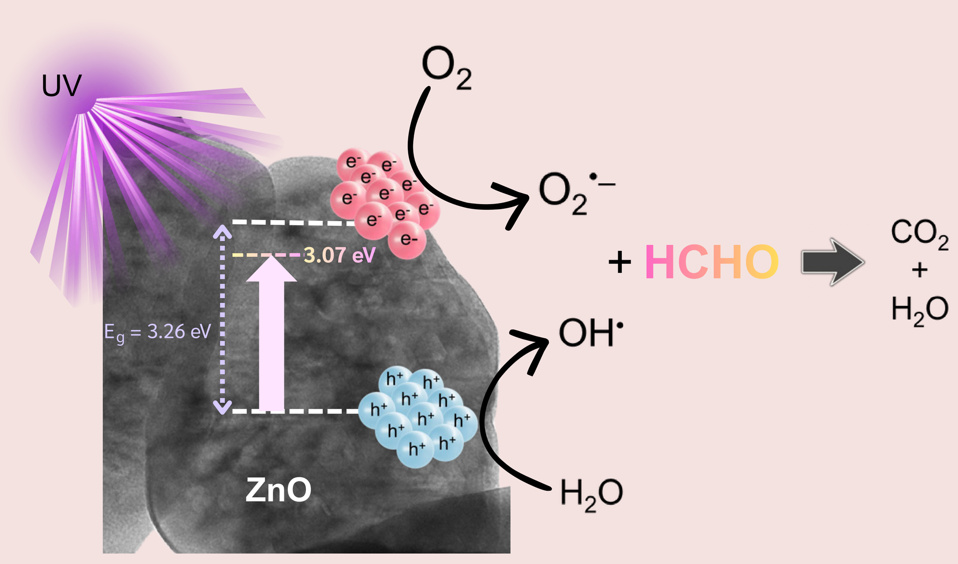


Figure 7. Photocatalytic degradation mechanism of formaldehyde.

$ZnO \xrightarrow{h\nu} e^- + h^+$

$h^+ + OH^- \longrightarrow {}^{\cdot}OH$

$h^+ + H_2O \longrightarrow {}^{\cdot}OH + H^+$

$e^- + O_2 \longrightarrow {}^{\cdot}O_2^-$

$HCHO + {}^{\cdot}OH \longrightarrow {}^{\cdot}CHO + H_2O$

${}^{\cdot}CHO + {}^{\cdot}O_2^- \longrightarrow HCO_3^- \xrightarrow{+H^+} HCOOOH \xrightarrow{+HCHO} HCOOH$

$HCOOH \xrightarrow{-H^+} HCOO^- \xrightarrow{{}^{\cdot}OH} H_2O + {}^{\cdot}CO_2^-$

$HCOO^- \xrightarrow{+h^+} H^+ + {}^{\cdot}CO_2^-$

${}^{\cdot}CO_2^- \xrightarrow{[O],\ {}^{\cdot}OH,\ h^+} CO_2$

Conclusions

1. High-purity ZnO NPs were successfully synthesized via a green synthesis approach using roselle extract, exhibiting an irregular morphology.
2. The photocatalytic degradation performance of formaldehyde (HCHO) under UV irradiation demonstrated that the c-ZnO-15% sample exhibited the highest activity, achieving complete (100%) degradation of HCHO within 40 minutes
3. The photocatalytic activity was controlled by surface structural features rather than by bulk crystallinity, and RDB-PAS proved sensitive to changes in the concentration of active surface structures across different synthesis routes.

Acknowledgment

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RDB-PAS Guide