



## Microwave-assisted Impregnation of Zinc Metal Ions on Surface of Quenched Pulverized Shrimp Shell Waste

Dwindrata Basuki Aviantara<sup>1,3\*</sup>, Fuzi Suciati<sup>1</sup>, Gunawan Hadiko<sup>2</sup>, Nastiti Siswi Indrasti<sup>3</sup>, Mohamad Yani<sup>3</sup>

<sup>1</sup>Indonesian National Research and Innovation Agency (BRIN), Geotech Puspiptek Serpong, Banten Province, 15314, Indonesia

<sup>2</sup>Ministry of Industry, Machinery Industrial Center, Sangkuriang 12 Bandung, West Java Province, 40135, Indonesia

<sup>3</sup>Department of Agroindustrial Technology, Faculty of Agricultural Engineering and Technology, IPB University, Raya Dramaga Street, Bogor, West Java, 16680, Indonesia

**Abstract.** This study evaluated the effects of microwave irradiation on the impregnation efficiency of zinc onto pulverized shrimp shell waste. Prior to impregnation, the pulverized shrimp shell was heat treated at 350, 450, and 550 °C for 1, 2 and 3 hours. After each treatment, the treated pulverized shrimp shell was immediately quenched in liquid nitrogen. Microwave-assisted impregnations were carried out at 80 °C for 10, 20, and 30 minutes and at 50% and 100% of the maximum delivery power rate of the laboratory microwave equipment. The resulting impregnated solids were characterized via TGA, DTA, FTIR, SEM, and XRD. Semi-quantitative measurement of the impregnated zinc was determined based on XRF analysis. The BET analysis of the original pulverized shrimp shell waste provided a surface area of 1.273 m<sup>2</sup>/g and achieved 30.423 m<sup>2</sup>/g after 550 °C for 3h heating treatment. An increase in crystallinity index was detected as the pulverized shrimp shell waste was exposed to higher and prolonged heating temperatures. Statistical analysis showed no significant difference ( $p = 0.05$ ) in impregnated zinc levels among the heat-treated pulverized shrimp shells for the same power output of microwave energy. However, a significant difference was obtained between 50% and 100% power output, of which the latter can impregnate two-fold higher levels of zinc than the former. The results of this study concluded that the microwave-assisted technique might potentially be applied for metal impregnation for the preparation of heterogeneous catalysts, and the power strength of the microwave plays a prominent role in metal impregnation.

**Keywords:** Impregnation; Microwave-assisted; Shrimp shell waste; Quenching; Zinc metal

### 1. Introduction

Crustacea are the most diverse groups of aquatic organisms that occupy a wide variety of habitats from the shoreline to the deep ocean and freshwater, where in some cases onto land for part of their life history (Penn, 2019). Crustacean species contribute to approximately 14.5 million tons (about 8%) of the total world supply of fish per annum, of which around half of this production is from the harvesting wild stocks, with major output from extensive and semi-intensive systems of tropical shrimps (Villareal and Juarez, 2022;

\*Corresponding author's email: [aviantara@rocketmail.com](mailto:aviantara@rocketmail.com), Tel.: 081119333604  
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Penn, 2019). As a result of their economic value, crustacean fisheries, particularly shrimps, are generally heavily exploited worldwide.

Both shrimp and shrimp products are widely consumed all over the world. Thus, it is not surprising that crustacean aquaculture is the largest seafood production sector around the globe. According to IMARC the global shrimp production in 2020 achieved 5.03 million tons (IMARC, 2020) and is expected to arrive at 7.28 million tons by 2025 when the shrimp market turnover value is expected to achieve 67.6 billion US dollars (Nirmal *et al.*, 2020) if compound annual growth rate (CAGR) of 6.1% is assumed. More than 80% of global shrimp production is from Asia (Mao *et al.*, 2017), where Asian countries contributed more than 2.5 million tons of the estimated production of marine shrimp (Suryawanshi and Eswari, 2021; Anderson, Valderrama, and Jory, 2019; FAO, 2017). Shrimp farming and processing plants produce the largest seafood industry worldwide due to their high demand and market value. From this, the shrimp processing industry produced 50 – 60% waste of the catch volume (Nirmal *et al.*, 2020), equal to 6 – 8 million tons of shrimp shell waste per year (Zhao *et al.*, 2021). The process of producing frozen shrimp creates remnants in the form of shrimp waste. Before being put into cold storage, the shrimp are peeled to remove the head, tail, and exoskeleton (Junianto *et al.*, 2021). At present, crustacean-based food industries are creating environmental problems as the leftover crustacean shell wastes are ubiquitous and produce unpleasant odors as such posing environmental and health risks to living organisms (Padida *et al.*, 2021). Traditionally the generated shrimp wastes are used for animal feed (Judhaswati and Damayanti, 2019; Has *et al.*, 2018; Hilkiyas, Suprijatna, and Ondho, 2017) or are processed for other products such as fermented paste (Latifah and Ria, 2016), shrimp crisp (Asmiarsari *et al.*, 2016) and Asian traditional food (Harmayani *et al.*, 2019). Rather advanced utilization of shrimp shell wastes includes shrimp-based compost (Rusmini, Manullang, and Daryono, 2017) and carotenoid and chitin isolation (Junianto *et al.*, 2021; Zhao *et al.*, 2019). From the latter substance can be derived various products such as glucosamine (Hardoko *et al.*, 2017; Mojarrad *et al.*, 2007), chitosan (Narudin *et al.*, 2022; Pakizeh, Moradi, and Ghassemi, 2021; Islam, Khan, and Alam, 2017), adsorbent (Salawu, Han, and Adeleye, 2022; Zein *et al.*, 2022; Shahrin *et al.*, 2021; Cahyaningrum and Amaria, 2005), bioplastics (Dasumiati, Saridewi, and Malik, 2019) and several products for biomedical and pharmaceutical purposes (Satitsri and Muanprasat, 2020; Usman *et al.*, 2018; Kusriani *et al.*, 2014; Park and Kim, 2010) including advanced utilization for encapsulation of quantum dots (Lim *et al.*, 2021). Furthermore, wide applications of shrimp shells as biomaterials for catalysis in biodiesel production have been reported (Kishore *et al.*, 2021; Suryawanshi and Eswari, 2021). The usual method for catalyst preparation is incomplete carbonization, followed by metal loading (Yang, Zhang, and Zheng, 2009). On the other hand, the utilization of shrimp shells in the catalysis of environmental pollutants decontamination is still lacking. This paper presented the possibility of synthesizing heterogeneous catalysts from quenched shrimp shell waste for environmental protection purposes using the metal impregnation technique with the assistance of microwave irradiation to accelerate the process. The technique has been reported as well for other purposes such as surfactant production (Qadariyah *et al.*, 2022), synthesis of fatty acids (Maulida *et al.*, 2020), and also alkaline delignification process (Harahap *et al.*, 2019).

## 2. Methods

### 2.1. Shrimp Waste Pulverization

The shrimp shell waste was obtained from a local company, Wirantono Baru Ltd., located in Balaraja Regency, Province of Banten. The current core business of the company is exporting frozen peeled shrimp from Indonesia to Japan, European countries, and South

American countries. Initially, the obtained wet shrimp waste was soaked and washed with clean water and then followed by air-dry. The air-dried shrimp shell waste was then pulverized into particles of size 170 mesh (approximately particle diameter 90 μm) and stored in a polytetrafluoroethylene box.

2.2. Heating and Quenching

Prior to impregnation with zinc metal ions, the pulverized shrimp shell waste was heated in an electrical furnace at temperatures 350, 450, and 550°C for 1, 2, and 3 hours. After that, the hot powder was immediately quenched in liquid nitrogen and stored in a polytetrafluoroethylene box until used. The effect of heating treatments on the physical structure of the pulverized shrimp shell waste at the aforementioned temperature range was analyzed using XRD Diffractometer Rigaku Miniflex 600.

2.3. Preparation of Zinc Stock Solution

Stock solution for zinc to be impregnated onto solid shrimp shell waste was prepared by dissolving 41.96 g of Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O of pure analytical grade (purchased from Merck) in aquadest until volume 1,000 mL in a volumetric flask. This will contain 12.5 g zinc per liter of stock solution. For the impregnation step, the solid quenched shrimp shell waste (in g) to zinc solution (in mL) ratio was 1 to 10; as such, the mixture will contain 12.5% of zinc metal ions loading in bulk solution.

2.4. Impregnation

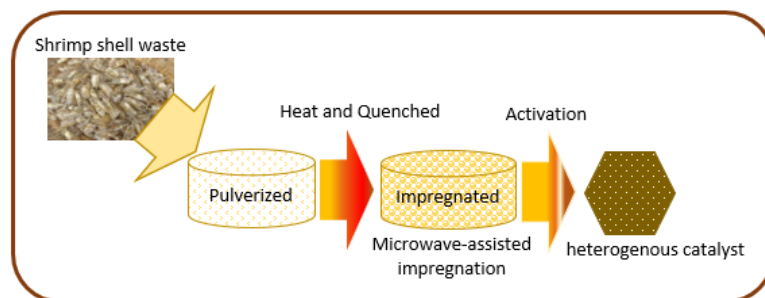
The process of impregnation was carried out using a MARS Xpress microwave. The MARS Xpress microwave is capable of delivering maximum power strength at 1800 W. For the purpose of the experiments, the power delivered into the impregnation chamber was varied at 50% and 100% of the maximum power, where the impregnation temperature was held at 80°C. The duration of the impregnation process also varied at 10, 20, and 30 minutes. Two and a half grams of quenched pulverized shrimp shell waste was put into the MARS Xpress sample holder and then added with 25 mL of zinc stock solution. Table 1 shows experimental parameters, namely heating temperature, heating duration, and impregnation time, of which each parameter is varied at three levels. Responses as levels of impregnated zinc for both 50% and 100% of microwave power strength were evaluated through the Box-Behnken design of the experiment.

**Table 1** Pulverized shrimp shell waste experimental design

Treatment	Symbol	Unit	Level		
Heating temperature	T <sub>H</sub>	°C	350	450	550
Heating duration	D <sub>H</sub>	hour	1	2	3
Impregnation time	D <sub>I</sub>	minute	10	20	30

2.5. Solid Characterization

In order to characterize pulverized shrimp shell waste before and after treatments then, several tools assessment that includes X-ray diffraction (Rigaku Miniflex 600), X-ray fluorescence (Energy Dispersive Rigaku Nex CG), scanning electron microscopy (using Thermo Phenom ProX), thermal gravimetry (Perkin Elmer TGS-2 Thermogravimetric Analyser), differential thermogravimetry (Shimadzu DTG-60H), and Fourier transform infrared (Bruker Tensor II FTIR) are utilized. Figure 1 presents a general scheme to prepare pulverized shrimp shell waste followed by microwave-assisted impregnation.

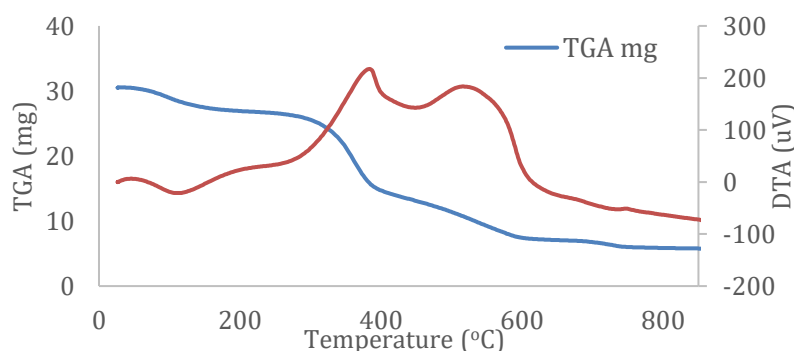


**Figure 1** Preparation of impregnated pulverized shrimp shell waste

### 3. Results and Discussion

#### 3.1. Thermogravimetric Profile

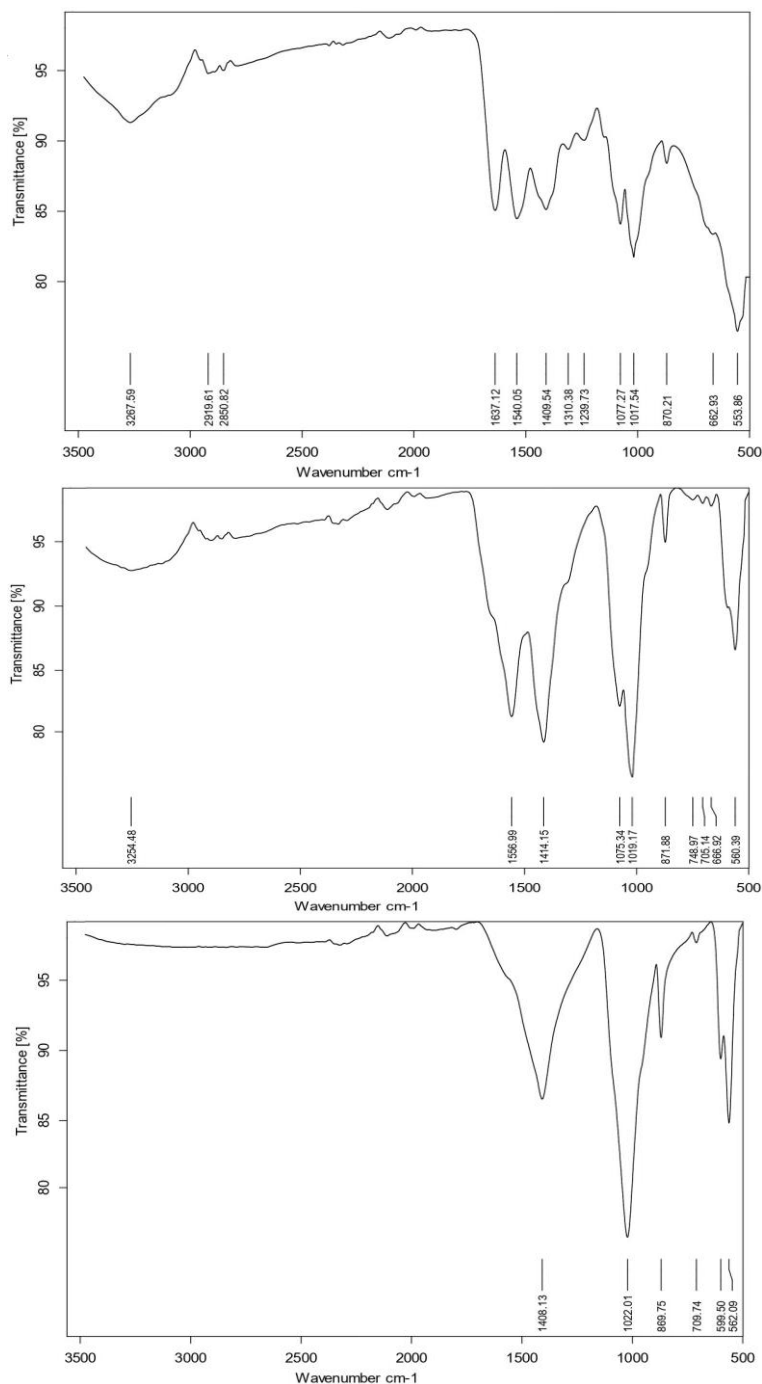
The thermogravimetric profile of the pulverized shrimp shell waste is shown in Figure 2. As can be seen from the graph, decreasing weight (blue line) in the range from ambient to 150°C could be attributed from releasing of the adsorbed water as well as volatile matters. Furthermore, significant weight reduction in the range of 300 – 400°C and 400 – 600°C might be attributed to the decomposition of residual and release of proteins and the acetyl group of chitin (Biondo *et al.*, 2015). In addition, the pattern of the differential thermal analysis (DTA) graph, the red line, demonstrates the presence of two exothermic peaks at around 390 and 525°C. Such the aforementioned two peaks are similar to the obtained DTA profile from another study of pure chitin (Vlaev, Georgieva, and Tavlieva, 2015).



**Figure 2** Thermogravimetric profile of pulverized shrimp shell waste.

#### 3.2. Fourier Transform Infrared Analysis

Results of the infrared peaks spectra for the original pulverized shrimp shell waste, heat treatment at 350°C for 1 hour, and at 550°C for 3 hours are presented in Figures 3a to 3c consecutively. As shown in Fig. 3a, the broad absorption of infrared energy between 3500 and 3200  $\text{cm}^{-1}$  could be attributed to the vibration of N–H groups and O–H bond stretching of a water molecule present in the pulverized shrimp shell waste. Overlaying Figures 3a to 3c recognized that several absorption peaks decreased when heating temperatures increased. Such decreased peaks could be attributed to releasing the adsorbed water, volatile matters, decomposition of residual, and release of proteins and acetyl group of chitin, as discussed previously (Biondo *et al.*, 2015). Therefore, absorption peak changes are in accordance with the TGA profile, as shown in Figure 2.

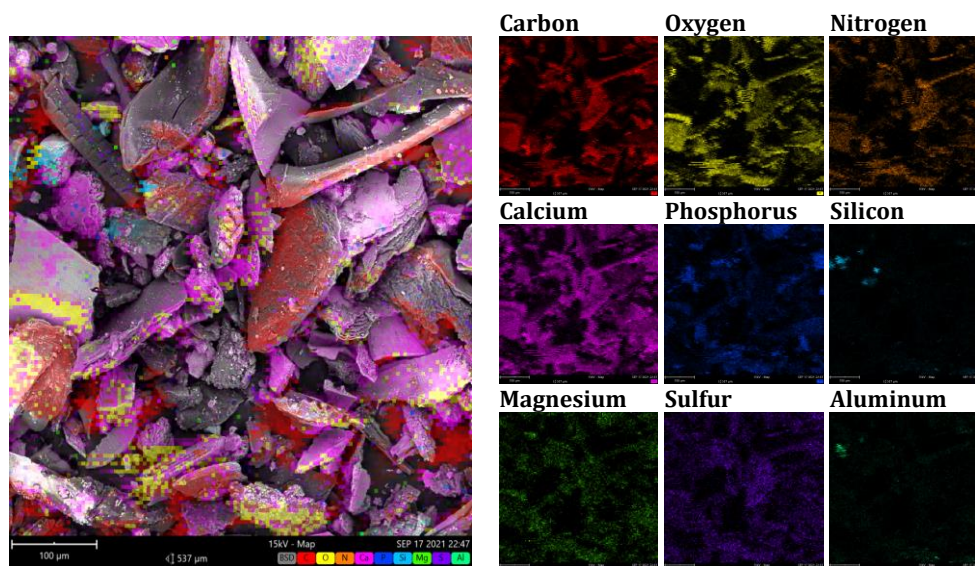


**Figure 3** FTIR peaks of original pulverized shrimp shell waste (a), quenched pulverized shrimp shell after 350 °C heat treatment for 1 h (b), and after 550 °C heat treatment for 3 h (c)

### 3.3. SEM Characterization

Prior to the heating process surface of the pulverized shrimp shell waste was analyzed using Scanning Electron Microscopy (SEM) Phenom as presented in Figure 4. As can be seen from the figure, a highly irregular morphology of the materials was recognized. Such an irregular morphologic with a size ranging from a few to hundred microns could be correlated with the chitin component as the pattern was similar to the chitin study (Nguyen *et al.*, 2022). The prominent visual colors of pink, red, and yellow demonstrate that the pulverized shrimp shell materials are prominently composed of calcium, carbon, and

oxygen elements. This is unsurprising as the major component of shrimp shells is calcium carbonate (Gbnedor *et al.*, 2016).



**Figure 4** SEM result of pulverized shrimp shell waste and its mapping color

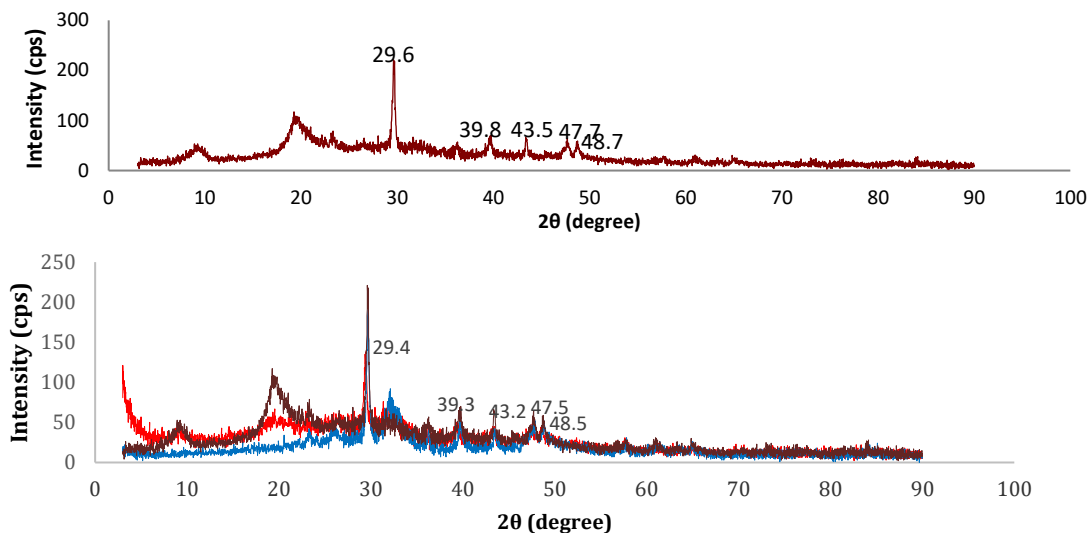
### 3.4. XRD Characterization

Figure 5 (above) demonstrates XRD peak patterns for pulverized shrimp shell waste prior to the heating process. As can be seen on the graph, peaks for  $2\theta$  appear at 29.6, 39.8, 43.5, 47.7, and 48.7. The diffractogram peaks resemble calcium carbonate, which is in accordance with SEM findings as described previously.

Comparing the obtained XRD peak patterns with a diffractogram of standard calcite mineral (Render *et al.*, 2016) as well as another study (Gbenebor *et al.*, 2017) demonstrated that calcite-type calcium carbonate dominates the mineral content of shrimp shell waste.

Figure 5 (below) is presented superimposed patterns of diffractogram peaks of the quenched pulverized shrimp shell waste after the heating process at 350 °C for 1 h (red line) and 550 °C for 3 h (brown line). Based on Figures 5 and 6, the calculated crystallinity index is presented in Table 2. As demonstrated by Table 2, increasing the temperature and prolonging the heating treatment is accompanied by an increase in crystallinity index as well as an increasing the surface area.

Several other researchers also observed similar effects in increasing crystallinity along with heat treatments using inorganic (Zaid *et al.*, 2019) or organic (Yusuf *et al.*, 2022) solid materials. A similar phenomenon in the increase of crystallinity was observed in a study of quenching temperature effects on inorganic solids (Shi *et al.*, 2012; Yun *et al.*, 2004). It is suspected that upon heating between 375 and 425 °C and between 500 and 550 °C (see Figure 2), the glassy state of the pulverized shrimp waste sample exhibit exothermic transitions that could correspond to the formation and subsequent perfection of secondary crystallization (Polaskova *et al.*, 2019), or formation of mesomorphic states (De Rosa *et al.*, 2003), i.e., a mesophase between a crystalline solid and an isotropic liquid where the molecules are separated in parallel layers of quasicrystalline order (Li, 2008) through diffusion or dissolution process (Mark *et al.*, 2004), that let in increasing of the crystallinity index. Also, more than 7-fold of the surface area increment (from 3.962 to 30.43 m<sup>2</sup>/g) of the pulverized shrimp shell waste was obtained when treatment of 550 °C for 3 hours is compared to 350 °C for 1 hour.



**Figure 5** XRD patterns of original (above) pulverized shrimp shell waste after heating treatments (below) at 350 °C for 1 h (red line) and 550 °C for 3 h (blue line).

**Table 2** Physical properties of shrimp shell before and after heating treatment.

Treatment	Crystallinity Index	Surface Area (m <sup>2</sup> /g)
Without heating treatment	67	1.273
350 °C for 1 h	77	3.962
550 °C for 3 h	89	30.43

### 3.5. XRF Semi-Quantitative Analysis

Presented in Table 3 are the results of the semi-quantitative elemental determination of the original pulverized shrimp shell waste using X-ray Fluorescence (XRF) analysis. As can be seen from the table, the original pulverized shrimp shell waste contained 12.5% of calcium and a very low level of zinc at 0.0059%. The big five detected elements in decreasing proportion are calcium (Ca), phosphorus (P), sodium (Na), silicon (Si), and magnesium (Mg) consecutively. Table 4 presents the results of microwave-assisted zinc impregnation onto the pulverized shrimp shell waste's surface. As can be seen in the table, several other metals commonly found in the shrimp shell are also reported, in addition to measured zinc levels.

As shown in Table 4, levels of impregnated zinc on the surface of the pulverized shrimp shell waste are in the range of 3.09 – 3.81% relative to the solid powder mass if 100% of microwave power strength was applied. This corresponded to 25 – 30% efficiency in impregnation. This low efficiency might be attributed to the coarse particle size of the pulverized shrimp shell. Another study is capable of obtaining a higher porous powder surface area using a 65 mesh screener pore size instead of 170 mesh, as used in this study. The impregnation power decreased at a 50% rate of power strength, provided levels of impregnated zinc were in the range of 0.86 – 2.04%. On average, from the results, altering the microwave power strength from 100% to 50% resulted in the latter being capable of providing levels of impregnated zinc half only than the former.

Results of the Box-Behnken evaluation of the controlled experimental parameters to response impregnated zinc onto pulverized shrimp shell waste at a rate of 100% and 50% of microwave energy power strength are presented in Table 5. It can be inferred that the combination of treatments (heating temperature, heating time, impregnation time) has no significant effects on the response (levels of impregnated zinc onto the surface of pulverized shrimp shell waste) based on threshold  $p = 0.05$  significant probability for each rate of microwave power strength. However, applying paired test using Minitab software (Table

4) for measured zinc levels for P = 100% and P = 50% resulted in a significant difference at  $p = 0.05$  probability. The output of the Minitab calculation for the paired test is presented in Table 6. Thus, from the results, it is confirmed that the power strength of microwave irradiation plays a prominent role in controlling levels of impregnated zinc compared to the combination of heating temperature, heating time, and impregnation time.

**Table 3** Elements weight % of original pulverized shrimp shell

Element	Weight (%)	Element	Weight (%)
Mg	0.4820	Ni	0.0014
Al	0.3330	Cu	0.0031
Si	0.5300	Br	0.0680
P	1.8500	Rb	0.0014
S	0.3680	Sr	0.1470
Cl	0.0787	Sn	0.0042
K	0.1470	I	0.0022
Ti	0.0185	Pb	0.0006
Cr	0.0047	Na	0.7040
Mn	0.0141	Ca	12.500
Fe	0.2230	Zn	0.0059

**Table 4** XRF semi-quantitative measurement of impregnated zinc onto the surface of pulverized shrimp shell waste.

Treatment			Mass proportion (%)									
T <sub>H</sub> (°C)	D <sub>H</sub> (h)	D <sub>I</sub> (min.)	P = 100%					P = 50%				
			Zn	Ca	Mg	Na	P	Zn	Ca	Mg	Na	P
350	1	20	3.09	14.6	0.661	1.34	2.81	0.902	16.2	0.887	1.70	3.71
350	3	20	3.54	17.9	0.786	1.62	3.14	0.856	18.6	0.851	1.19	3.60
550	1	20	3.81	21.6	0.926	2.53	3.72	1.43	23.2	1.08	1.07	4.55
550	3	20	3.68	24.4	1.160	4.04	4.43	2.04	26.9	1.01	1.95	5.10
350	2	10	3.46	18.0	0.789	1.58	3.78	1.82	19.3	0.898	1.39	3.96
350	2	30	3.31	18.2	0.859	2.12	3.36	1.70	20.1	0.931	1.32	4.07
550	2	10	3.40	22.6	1.02	3.29	4.53	1.29	24.4	1.10	1.14	4.72
550	2	30	3.50	21.9	1.06	2.83	4.22	1.56	23.1	1.04	1.32	4.57
450	1	10	3.64	20.8	0.877	2.07	3.99	1.24	23.3	1.02	1.36	4.47
450	1	30	3.40	22.2	1.140	3.85	4.00	1.93	24.6	1.06	1.46	4.70
450	3	10	3.62	21.5	0.975	2.84	4.31	1.60	23.1	1.03	1.13	4.53
450	3	30	3.40	22.6	1.190	4.42	4.33	1.87	23.5	1.08	1.38	4.84
450	2	20	3.39	19.8	0.945	2.11	3.47	1.23	22.2	1.06	1.34	4.27
450	2	20	3.71	19.8	0.917	2.72	3.98	1.56	21.2	1.02	1.67	4.21
450	2	20	3.70	19.6	0.968	2.70	3.67	1.87	27.4	1.01	1.91	5.16

**Table 5** Box-Behnken analysis for impregnation at P = 100% and P = 50%

Term	<i>p</i> -value at P = 100%	<i>p</i> -value at P = 50%
Constant	0.000	0.041
Heating temperature (°C)	0.952	0.921
Heating time (hour)	0.879	0.524
Impregnation time (minute)	0.442	0.536
Heating temperature (°C) *Heating temperature (°C)	0.421	0.531
Heating time (hour)*Heating time (hour)	0.891	0.717
Impregnation time (minute) *Impregnation time (minute)	0.348	0.439
Heating temperature (°C) *Heating time (hour)	0.175	0.497
Heating temperature (°C) *Impregnation time (minute)	0.526	0.682
Heating time (hour) *Impregnation time (minute)	0.959	0.659



**Table 6** Minitab output for a paired test of P = 100% and P = 50%

Descriptive Statistics				
Sample	N	Mean	StDev	SE Mean
%Zn (80C 100%P)	15	3.5100	0.1880	0.0485
%Zn (80C 50%P)	15	1.5265	0.3646	0.0941
Estimation for Paired Difference				
Mean	StDev	SE Mean	95% CI for $\mu$ difference	
1.9835	0.3595	0.0928	(1.7844; 2.1826)	
$\mu$ difference: population mean of (%Zn (80 °C 100%P) - %Zn (80 °C 50%P))				
Test				
Null hypothesis	$\mu_0$ : $\mu$ difference = 0			
Alternative hypothesis	$\mu_1$ : $\mu$ difference $\neq$ 0			
T-Value	P-Value			
21.37	0.000			

#### 4. Conclusions

Results of this study showed that no quenching nor impregnation contact time affects the levels of impregnated zinc. On the other hand, heating to a temperature up to 550 °C followed by subsequent quenching resulted in an increase in the degree of crystallinity as well as surface area. Furthermore, implementing microwave irradiation at full power strength 100% is capable of impregnating the zinc metal shell two-fold higher onto the pulverized shrimp's surface than at half power strength 50%. The microwave-assisted method may potentially be applied for zinc impregnation in preparing heterogeneous catalysts from shrimp shell waste. The power strength of microwave irradiation is the key parameter to be controlled to achieve a better impregnation process.

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