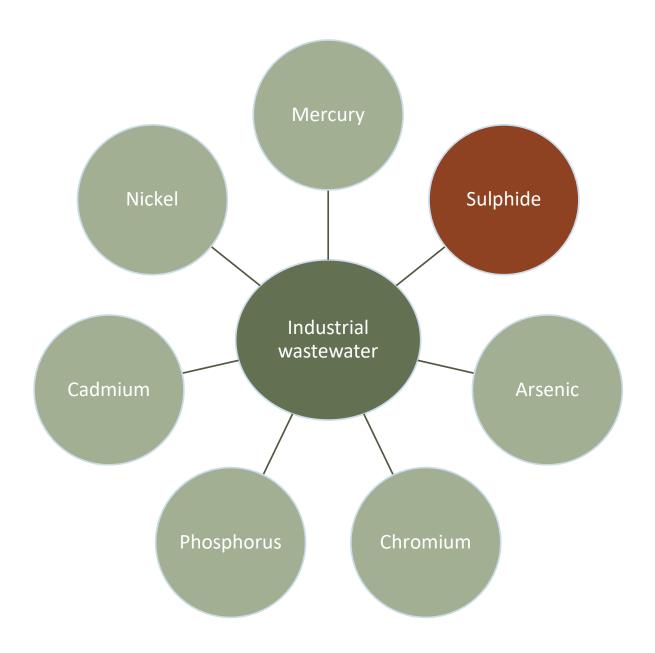
### The Potential Of Silica From Rice Husk Ash On Removal Of Sulphide In Wastewater

NURUL ALIA NABILAH BINTI DZOLKIFLE

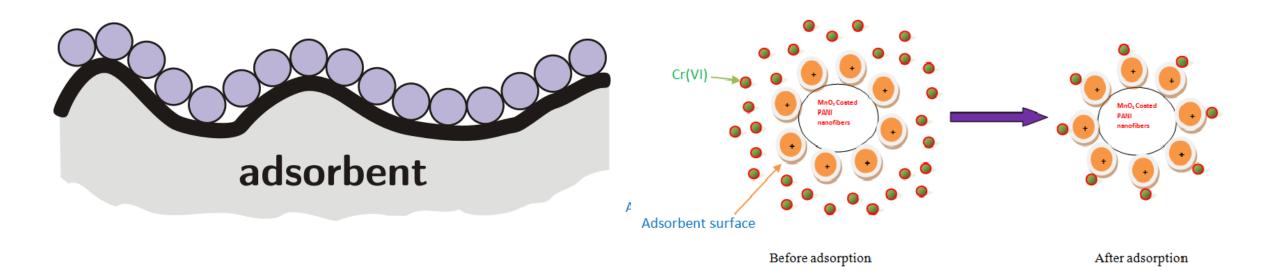
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# SULPHIDE

- Inorganic anion of sulphur derived from sodium sulphide or hydrogen sulphide
- Can be found in petrochemical industry wastewater
- High concentration of sodium sulphide can be produced during propylene production from propane dehydrogenation process
- Excess amount of sulphide can lead to:
  - corrosion of sewage pipes
  - high fish mortality
  - obnoxious odors

### **ADSORPTION**



Adsorption is a surface phenomenon that causes accumulation of atoms, molecules, ori Wang & Peng, 2009; Tareq et al, 2019 ons at the interface of two phases like solid and liquid or solid and gas.

Adsorption has been proved to be a simple and effective technique in treating wastewater effluent and its success basically depends on the efficiency of the adsorbents.

- $\rightarrow$  low cost maintenance
- $\rightarrow$  high efficiency adsorbents
- $\rightarrow$  easy and simple treatment process
- $\rightarrow$  require short treatment time
- → enable to remove a wide range of heavy metal contaminants



**RICE HUSK ASH (RHA)** 



CLAYS

#### **ADSORBENTS**

- Low-cost adsorbents can be easily obtained due to its abundance in nature and require a little modification to improve their adsorption capacity.
- Mostly made up of industrial wastes, agricultural wastes and natural materials
- Known as ion exchanger
- Natural zeolite → clinoptilolite, mordenite, natrolite, heulandite, chabazite, philipsite, analcime, laumontite and erionite
- Clay minerals → smectites (such as montmorillonite), kaolinite and micas
- Rice husk ash (RHA) → contain more than 90% of silica, good at eliminate inorganic contaminants due to high porosity and large surface area

### **OBJECTIVES & HYPOTHESIS**

- To investigate the potential of low-cost adsorbents such as natural zeolite, clay and RHA to remove sulphide (S<sup>2-</sup>) in industrial wastewater.
- To characterize the concentration of adsorbents, pollution loading and periods of contact time for maximum efficiency of the technology

Low-cost adsorbents such as zeolite, clay and RHA have the potential for removing S<sup>2-</sup> in industrial wastewater using adsorption treatment method.

### Production of Silica from RHA





200 g of RHA was added into 1.2 L of hydrochloric acid (HCl) solution. Heated up to 80°C and immersed overnight Filtered, washed with distilled water and dried up to 70°C for 1 hour and desiccated in a furnace at 700°C for 2 hours

# Characterization of Silica from RHA

- □ Particulate size analysis (PSA) was used by applying sonication process using probe sonicator with dilution level of 0.01 g/20 mL (range between 0 10,000 nm)
- Samples were observed using scanning electron microscope (SEM) for size and morphology
  - gold coating before being observed

Performed Fourier Transform Infrared (FTIR) measurements using FTIR spectrometer – to

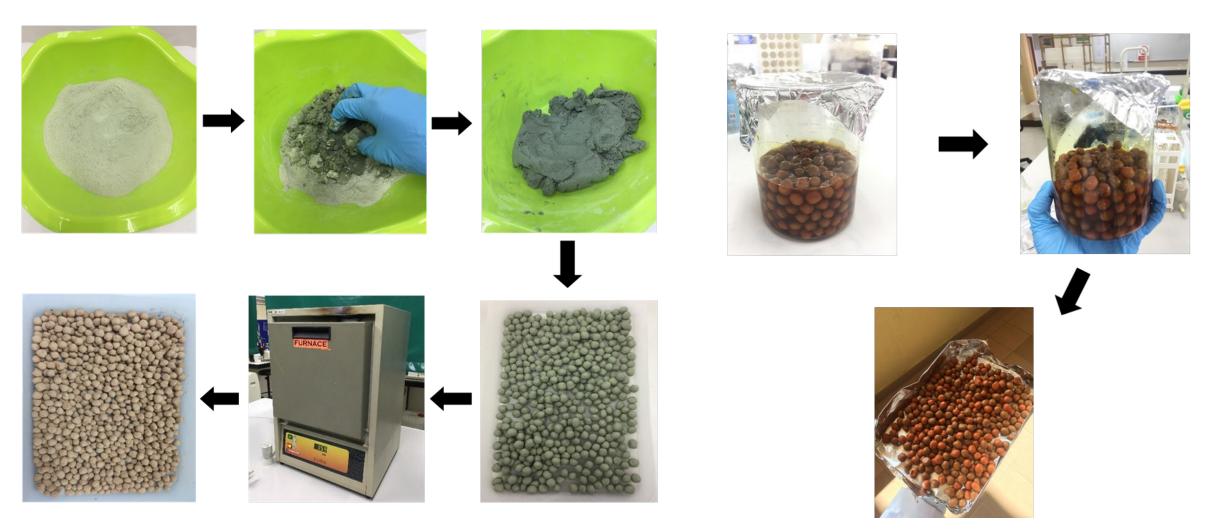
determine silica functional groups





#### PREPARATION OF TREATMENT BALLS

#### SURFACE MODIFICATION









Weight adsorbents by 1, 5, 10, 15 and 20 Prepare stock solution (100 ppm). Dilute into 20 ppm (fix concentration) in 100mL (triplicate) Insert adsorbents into each flask Put into incubator shaker with temperature at 40°C. Pipette 5 mL of solution from each flask. Check the absorbance (nm) using USEPA Methylene Blue Method. Check OD for 0, 60 and 120 minutes.







#### SULPHIDE CONTENT ANALYSIS





#### STATISTICAL ANALYSIS

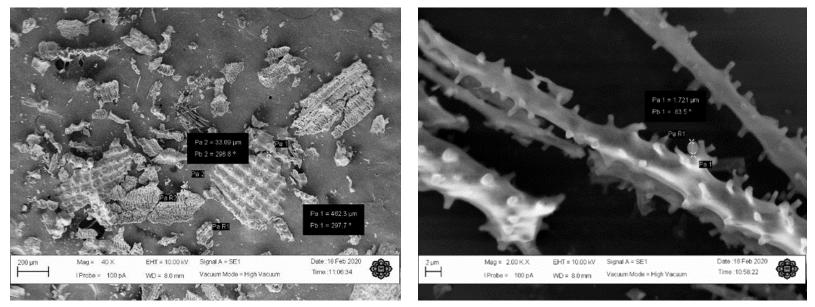
- Microsoft Excel recorded concentration of sulphide, calculated means and standard deviation, plotted using clustered columns
- Sigmaplot performed one-way ANOVA test

- Using US EPA Methylene Blue method (Sulphide 1 and 2 reagents)
- Absorbance measured using UV Vis Spectrophotometer – wavelength 665 nm

#### **RESULTS AND FINDINGS**

#### Production of nanosilica from RHA

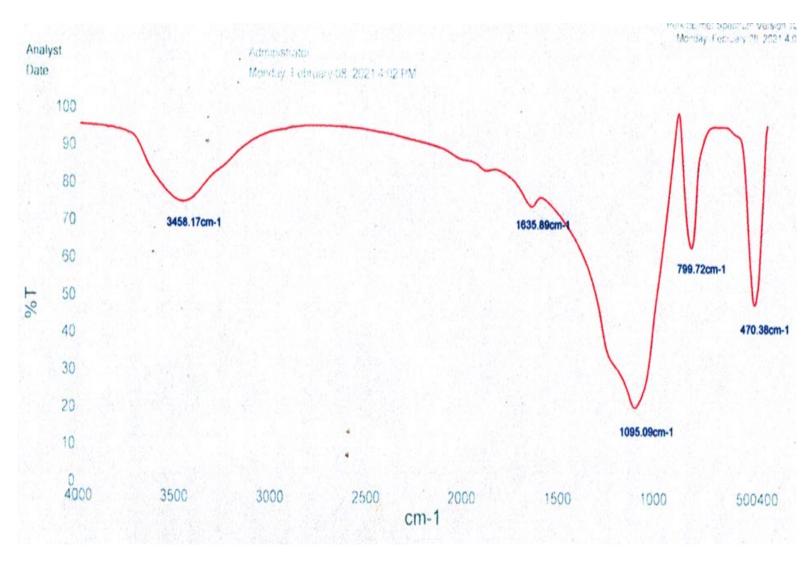
- 62% of silica was successfully recovered from RHA with around 37% weight loss
- Large clumps of particulate with a rough patterned texture at the outer epidermis
- Average size distribution of silica is around 0.9 2.0 μm
- Pa 1 and Pa 2 shows the particulates with large in size – diameter of 462.3 μm and 33.09 μm



The calcined RHA sample observed under SEM. A) Magnification level of 40 X, with scale of 200 μm. B) Magnification level of 2000 X, with a scale of 2 μm. A spheroid structure (Pa 1) is found attached to a fibre like structure with a diameter of 1.721 μm.

Sample	Average Size (µm)		
1	1.674		
2	0.9237		
3	1.190		

Average size distribution of silica extracted from RHA.



FTIR spectra of extracted RHA powder.

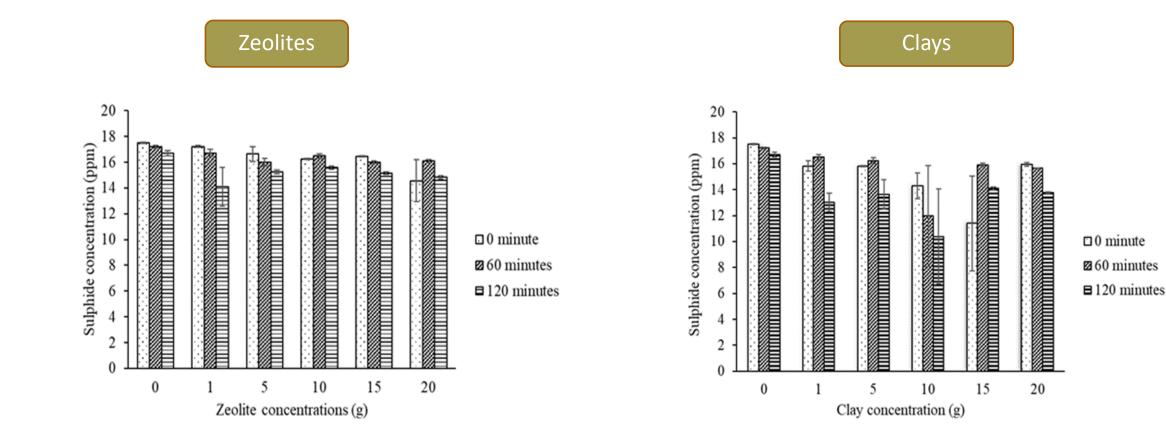
Absorption peaks present at 3458.17 cm-1, 1635.89 cm-1, 1095.09 cm-1, 799.72 cm-1 and 470.38 cm-1.

**B**road peak at 3458.17 cm-1 is primarily due to the presence of -OH bond stretching such as silanol hydroxyl groups (Si-OH).

Observed at 1635.89 cm-1 due to presence of impurities such as sodium and carbonate group (-COOH).

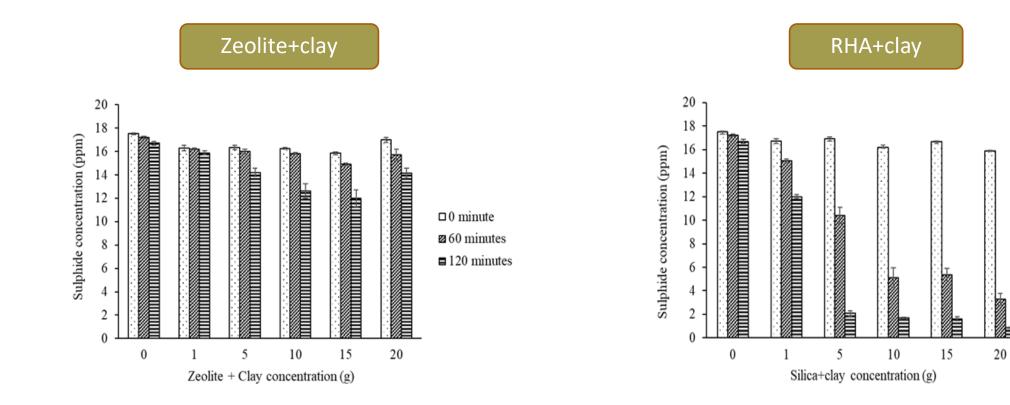
The peak at 1095.09 cm-1 represents functional groups of Si-O-Si.

799.72 cm-1 and 470.38 cm-1 are due to the presence of simple hydroxyl compound and Si-H respectively.



- Only 7 % of S2- was removed from wastewater samples treated with zeolite adsorbent after 120 minutes of treatment time (p>0.05)
- Efficiency of natural zeolites was better in removing cations rather than anions

- Able to remove up to 30 % of S2- in wastewater especially after treated for 120 minutes (p>0.05)
- The adsorption capacity of clay for anion removal is slightly greater than zeolites (7 % removal)



- Able to remove S2- more efficient compared to zeolite and clay itself with 16 % of S2- removal
- the concentration of adsorbents increased, the concentration of S2- in wastewater samples was also gradually decreased over treatment time (p<0.05)</li>

 94 % removal of S2- was recorded when 20 g of silica balls were tested on 20 ppm of sulphide for 120 minutes.

 $\Box 0$  minute

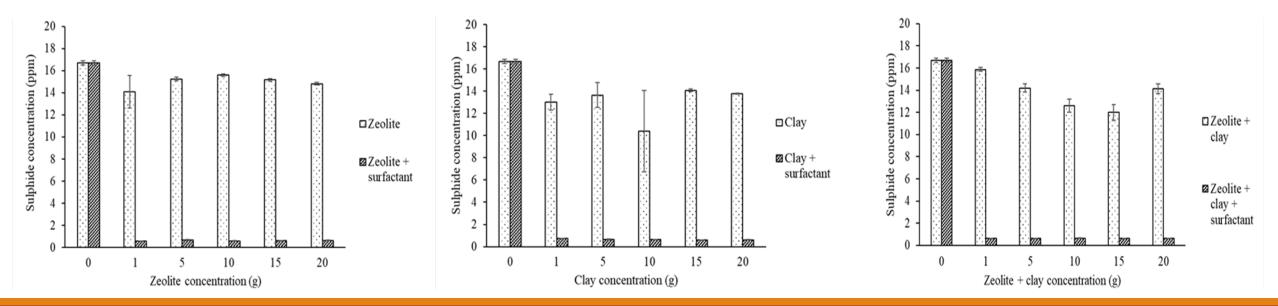
☑ 60 minutes

■120 minutes

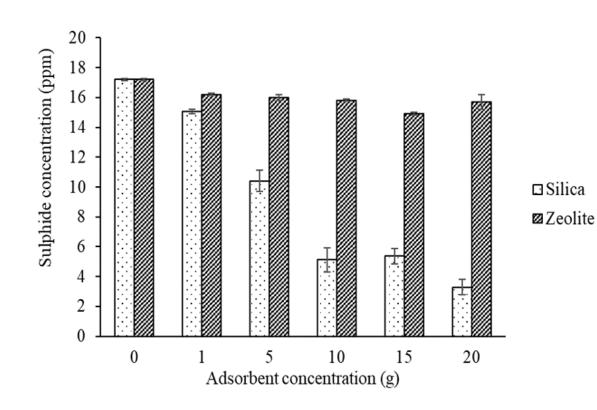
 More than 50 % of S2- removal was obtained when 10 g of silica balls was tested for 60 minutes

#### EFFECT OF CATIONIC SURFACTANT TREATMENT TOWARDS SULPHIDE REMOVAL BY ADSORBENTS

- FeCl3 was selected as cationic surfactant for the surface modification
- A significant reduction of S2- concentration in wastewater treated with modified zeolite compared to the unmodified ones where S2- was nearly removed within 120 minutes of treatment time (p<0.05)
- Due to the binding capacity of anions with cationic surface of modified zeolite and clay adsorbent as the surface of adsorbents was dominantly occupied with positively charged ions from FeCl3



#### COMPARISON BETWEEN THE PERFORMANCE OF ZEOLITE AND SILICA ON SULPHIDE REMOVAL



- Silica balls showed higher efficiency in reducing S2- concentration compared with zeolite with more than 50 % after 10 minutes of treatment (p<0.05)</li>
- 10, 15 and 20 grams of silica balls were able to remove more than 50 % of 20 ppm S2- levels after 60 minutes of treatment compared to zeolite
- Due to their larger surface area, higher porosity and greater adsorption capacity which able to eliminate more ions, and thus become an effective adsorbent in wastewater treatment

### REMOVAL RATE CONSTANT FOR S2- EXPRESSED AS FIRST ORDER EQUATION

$$\frac{-dC}{dt} = \frac{VmC}{Ks + C}$$

(mg <sup>-S</sup> L <sup>-1</sup> )	k (d <sup>-1</sup> )		Log (ge-qt)		R <sup>2</sup> values	
	Zeolite	Silica	Zeolite	Silica	Zeolite	Silica
1	-0.0033	0.04	1.177394	0.885721	0.9938	0.9756
5	0.0207	0.12	1.13115	0.64256	0.8502	0.9956
10	0.0319	0.14	1.037576	0.295187	0.905	0.9507
15	0.039	0.14	0.922075	-0.00876	0.9075	0.9578
20	0.022	0.15	0.965931	-0.39956	0.9654	0.9297

### CONCLUSION

- Low-cost adsorbents such as RHA offers the possibility for in situ removal of contaminants in industrial wastewater.
- Result indicates that silica has high potential in removing sulphide from wastewater without being
  need for further chemical treatment as compared to natural zeolite and clay which offers a greener
  approach in remediation of industrial wastewater.
- Further research on other parameters such as pH of wastewater must be investigated to ensure the water is neutral and safe before released into water sources.

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# THANK YOU