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Comparative Study on Natural and Commercial Coagulants: Treatment of Semiconductor Wastewater in Sludge Production and Removal of Heavy Metals

Mohd Omar Fatehah, Md. Sohrab Hossain, Tjoon Tow Teng

Abstract— The objective of this study is to determine the coagulation efficiencies on total solids (TS) removal from semiconductor wastewater by applying various locally available starches as natural coagulants. Two commercial coagulants such as alum [$Al_2(SO_4)_3 \cdot 18H_2O$] and polyaluminium chloride (PAC) were also utilized in this study in order to compare the coagulation efficiency with the natural coagulants. The EDX analysis of the raw semiconductor wastewater showed that it contained silica dioxide (SiO_2) with a concentration of 90%. It appeared that the natural coagulants employed in the study have similar coagulation characteristics with the commercial coagulants. However, the natural coagulants possess better metal adsorption capability than the commercial coagulants. A 3 level factorial experimental design was used in the Response Surface Methodology (RSM) analysis and indicated that starches are capable to remove TS from the semiconductor wastewater and the removal performance were almost similar to alum and higher than PAC.

Index Terms—Heavy metals, natural coagulants, response surface methodology, semiconductor wastewater, total solids.

I. INTRODUCTION

The semiconductor industry has progressively grown at a rapid pace in many countries around the world for the past several decades. The massive production of semiconductors which is widely used for computer parts, communication equipment, and electronic control devices has equally produced large amounts of wastewater that potentially poses a threat towards the environment [11]. The wastewater contains hazardous pollutants such as organic solvents, acids, bases, salts, heavy metals, fine suspended oxide particles and other organic compounds which are generated due to a large number of highly complex and delicate processes including silicon growth, oxidation, doping, photolithography, etching, stripping, dicing, metallization, planarization, cleaning, etc [24]. Various studies have been conducted on the treatment of semiconductor wastewater with different methodologies and emphasis. Reference [7], [16] investigated the application of dissolved (DAF) and dispersed (DiAF) air flotation to treat nanosized silica and fluoride-containing semiconductor wastewater. Chemical oxygen demand (COD) and turbidity removal were studied using electrocoagulation [5], [15] membrane technology with a three stage system was applied to reduce suspended solids [13] whereas treatment consisting a combination of physical, chemical and biological techniques were studied as well [14], [17], [18], [8].

Semiconductor wastewater, also known as CMP wastewater is highly turbid with a milky color due to its high slurry content [16]. Chemical and physical properties of CMP wastewater have been mentioned elsewhere [5]. Removal of TS particles has become a major concern. Studies have reported that chemical coagulant dosage is difficult in dealing with the nanosized particles in the wastewater. Adding calcium chloride ($CaCl_2$) or lime ($Ca(OH)_2$) for removal of fluoride will generate CaF_2 sludge. One of the major drawbacks of using $CaCl_2$ is that the precipitates are very fine and difficult to settle. The utilization of over doses of polyaluminium chloride (PAC) will only generate hazardous chemical sludge at higher chemical costs [6] which requires disposal as scheduled waste [1],[5]. Extended usage of chemical coagulants have disadvantages such as large dosage, low effect, high costs and toxicity [3], [5], [25]. Due to these reasons, this has lead us to search for a more economical, environmentally safe and natural polymer that may be feasible in treating the semiconductor influent. Therefore, starch has become a considerable interest as a natural coagulant to substitute commercial coagulant to treat the wastewater.

In recent years, natural polyelectrolytes of plant origin (i.e., *Moringa oleifera* seeds, *Cactus latifaria*, nirmali seed, mesquite bean, etc.) have been applied to treat industrial effluents as coagulants [2], [21], [25]. The coagulation performance of the natural coagulants has shown similar efficiencies as commercial coagulants in the clarification of wastewater [25]. Natural coagulants have the significant advantages over the commercial ones as they are available in abundance, cheaper, environmental friendly, multifunction, and biodegrades naturally in water purification [23]. In certain parts of the world, plant seeds have been utilized as coagulants. For example, *Moringa oleifera* is a kind of natural macromolecular coagulant that mainly grows in semi-arid, tropical, and subtropical areas, whereas cactus is known to grow in torrid zones [4],[25]. Other macromolecular polymers, i.e. starches are generally available in almost every continent in the world. Starch is a natural polymer that is

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categorized as a polyelectrolyte and can be proceed as a coagulant [20]. However, few studies have been conducted to treat CMP wastewater using locally available starches as natural coagulants. Sago, potato [20], and tapioca [9] flour was reported to treat semiconductor wastewater to reduce COD and total suspended solids (TSS) concentration.

The objective of this study is a consecutive step from previous preliminary studies [20], [9] in order to determine the feasibility of other locally available starches such as corn flour, flour, rice flour and glutinous rice flour as natural coagulants to treat semiconductor wastewater. The efficiency of the treatment was evaluated based on the reduction of heavy metals concentration and removal of TS. Two commercial coagulants such as alum [$Al_2(SO_4)_3 \cdot 18H_2O$] and PAC were also applied in this study with the same objective in order to compare the coagulation performance with the natural coagulants.

II. MATERIAL AND METHODS

Semiconductor wastewater was collected via composite sampling from a multi-national semiconductor plant, Advanced Micro Devices (M) Sdn. Bhd, located in Bayan Lepas, Pulau Pinang, Malaysia. The samples collected were labeled and stored at 4^oC. Natural polymers (corn, potato, tapioca, rice and glutinous rice flours) were purchased from the local market and applied as natural coagulants. Stock solutions were prepared for each natural coagulant at concentrations determined by the RSM. Analytical grade commercial coagulants, alum [$Al_2(SO_4)_3 \cdot 18H_2O$] and PAC were purchased from Sigma Aldrich.

Five hundred milliliters of the raw wastewater and the desired amount of coagulants were placed into a 500 mL beaker in a jar test apparatus (JLT6, VELP, SCIENTIFICA). The pH was adjusted to pH 12. The desired amounts of coagulants were added based on the design of experiment of response surface methodology (RSM). After adding the coagulant, the stirrer was set at 100 rpm with a mixing time of 5 minutes. The stirrer speed was then slowed down to 10 rpm and continued mixing for 15 minutes. The treated wastewater was allowed to settle for the desired duration of time according to the RSM design of experiment. The supernatant was then filtered using Whatman No. 1 filter paper (particle retention: 11 µm) and collected for TS analysis. The TS concentration was calculated according to standard APHA section 2540A and section 2540D. Scanning electron microscope (SEM) (Model-Leo Supra 50 VP) was used to observe the surface structure of coagulants before and after coagulation treatment. All the images were taken using working distance at 9 mm, and the acceleration voltage was 15.00kV. Energy dispersive X-Ray were used to analyze elemental composition in the sludge. Heavy metal concentrations i.e. arsenic, cadmium and zinc were measured using Atomic Absorption spectrophotometer (Model- Perkin Elmer, Model Analyst 700).

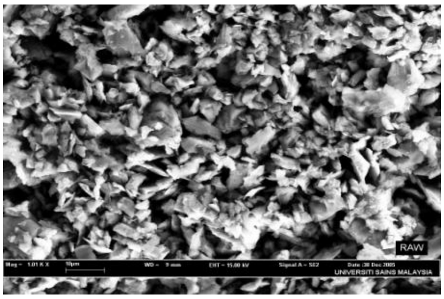
A Three Level Factorial design in the software of RSM was applied in the jar test coagulation study as mentioned by [20], [9]. With the same experimental design approach, the output of the interactions among the experimental factors were TS and heavy metals.

III. RESULTS AND DISCUSSION

A. *Sludge analysis of natural and commercial coagulants*

The structure of the silica particles and its characterization in raw semiconductor wastewater is shown in Fig 1. Fig.1 (a) shows the silica particles in the semiconductor wastewater in the formation of geometrical crystals. We observed that the silica particles in the semiconductor wastewater were dispersed for a long time like colloidal silica. Fig. 1 (b) displays EDX spectrum of the elemental composition of silica particles. From the spectrum band of silicon and oxygen, as presented in the EDX image, the particles were silicon dioxide (silica, SiO_2).

The SEM image of coagulated sludge after coagulation treatment using PAC is presented in Fig. 2(a). We found that the sludge was formed in a crystal shape. In Fig. 2(b), the EDX analysis shows that silica, sodium, and aluminium are present in the sludge. However, no regular shape was detected of the coagulated sludge upon using alum as a coagulant, as presented in Fig. 3(a). Fig. 3(b) shows the EDX analysis of the sludge shows the presence of silica, sodium and aluminium.



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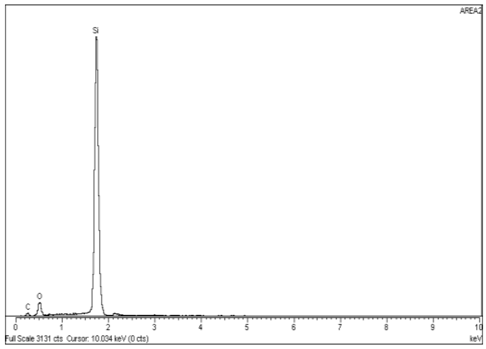

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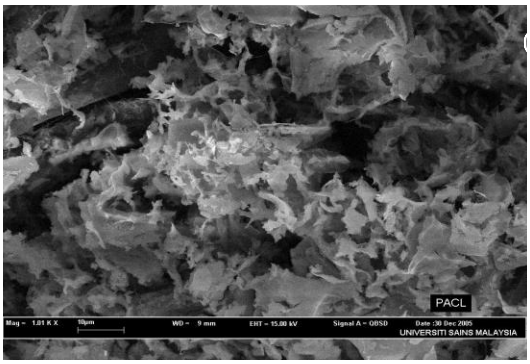
Fig.1. analysis of raw semiconductor wastewater sludge; (a). SEM micrograph; (b). EDX analysis.



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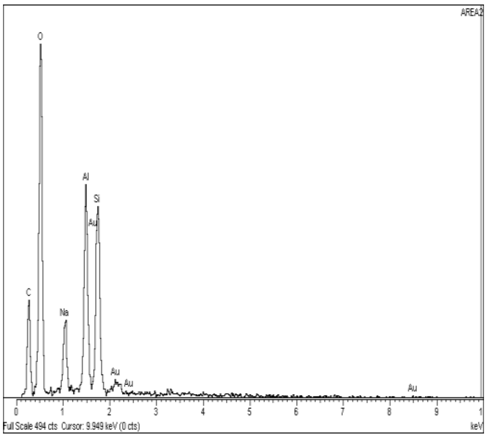

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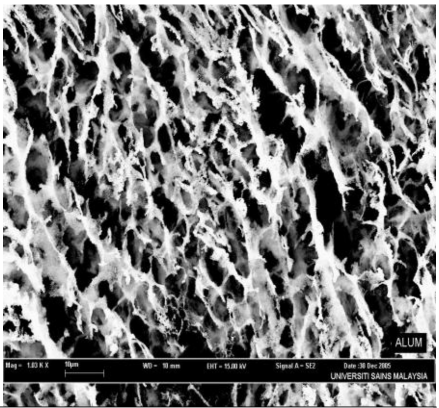
Fig. 2. (a) SEM image of the surface of the treated sludge using PAC as a coagulant; (b) EDX analysis of treated sludge.



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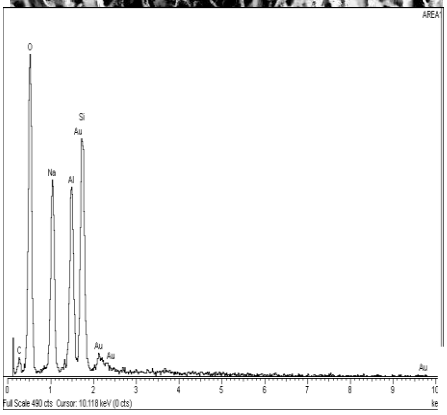

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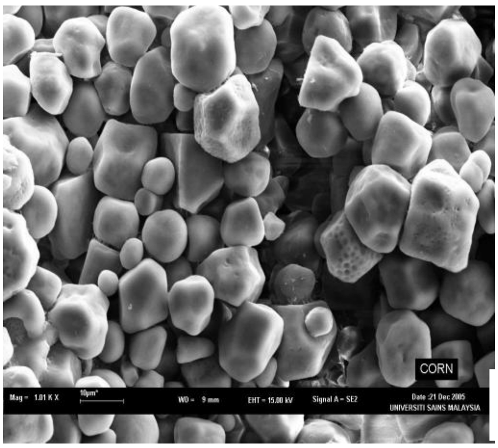
Fig. 3. (a) SEM image of the surface of the treated sludge using alum as a coagulant; (b) EDX analysis of treated sludge.



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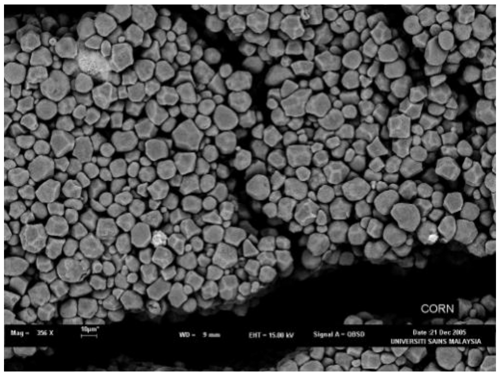

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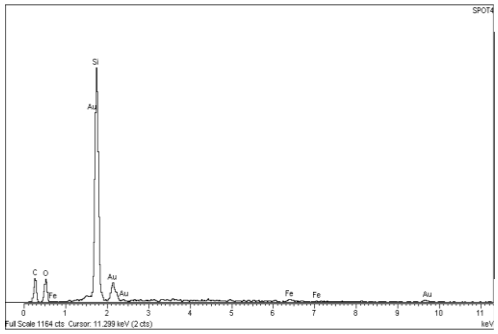

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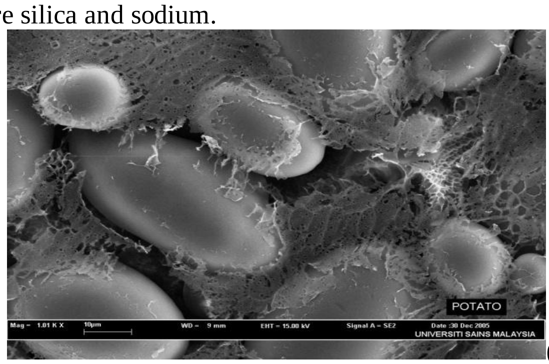
Fig. 4. (a) SEM image for the surface structure of corn flour; (b). SEM micrograph of filtered sludge using corn flour as a coagulant; (c). EDX analysis of the treated sludge.

Fig. 4(a) presents the SEM image of corn flour. We found that the surface of the sludge was covered with the coagulant. The shape of the corn starch was either polygonal or round. Fig. 4(b) shows the surface structure of the filtered sludge using corn flour as a coagulant. There are some elements present in the sludge. Fig. 4(c) shows the EDX analysis shows that the elements present on the surface of the sludge are silica and iron. However, the silica content found is higher than iron. Basically, the lower atomic number elements tends to absorb more electron, and the image of the element will be comparatively darker with the higher atomic number elements which tends to absorb less electron [10]. Fig. 5(a) shows the starch structure of the potato flour. We found that the molecular shape of the potato starch is oval and spherical. There are some empty spaces among the potato particles. Fig. 5(b) presents the SEM image of filtered sludge with potato flour after the coagulation process. We found that there are some contrast spots in the image, which indicates the presence of other elements in the sludge. Fig. 5(c) presents the EDX analysis indicates that the elements were silica and sodium.



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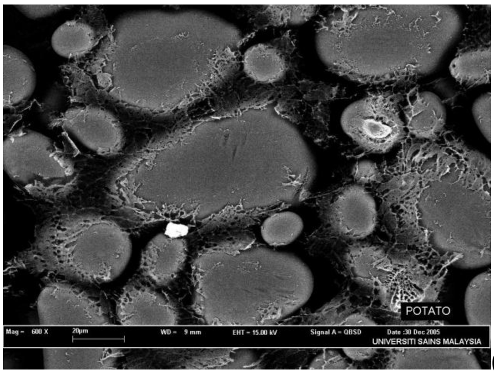

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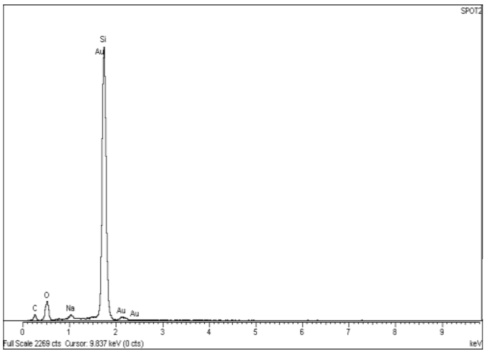

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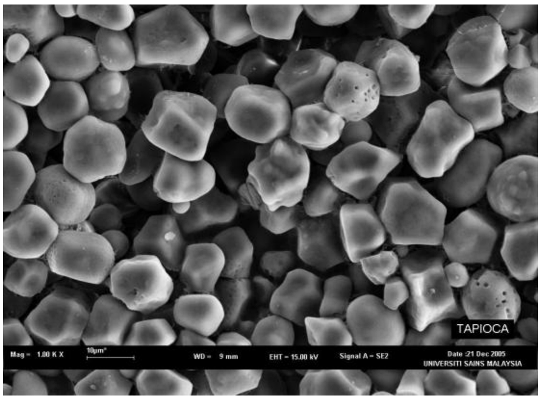
Fig. 5(a). SEM images of potato flour, (b). SEM image of filtered sludge using potato flour as a coagulant, (c). EDX analysis of the treated sludge.

The surface structure of tapioca flour is spherical and truncated in shape, as presented in Fig 6(a). In Fig. 6(b), contrast spots were identified, which proved the presence of elements in the tapioca starch coagulant. The EDX analysis shows the presence of sodium and aluminium on the surface of the coagulated sludge. However, the contrast spots on the surface of the sludge is most probably silicon particles as shown in Fig. 4(b). This is due to having higher atomic number to aluminium [10].

Fig. 7(a) shows the SEM image of rice flour. However, the image shows that the shape of the rice starch particles are in the form of polygonal, spherical, and compound granules. Fig. 7(b) is the SEM image of the filtered sludge after the coagulation treatment using rice flour as a coagulant. In Fig. 7(b), the existence of few contrast spots indicates the presence of other elements. The EDX analysis shows traces of zinc and sodium on the surface of the sludge as presented in Fig. 7(c).



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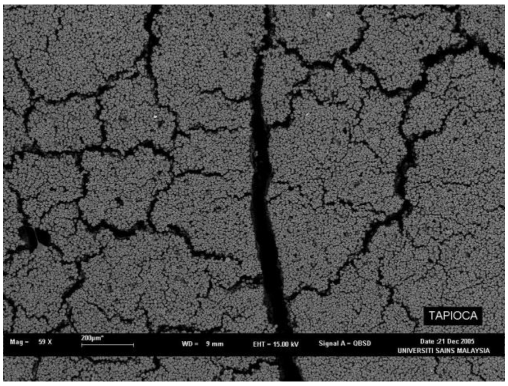

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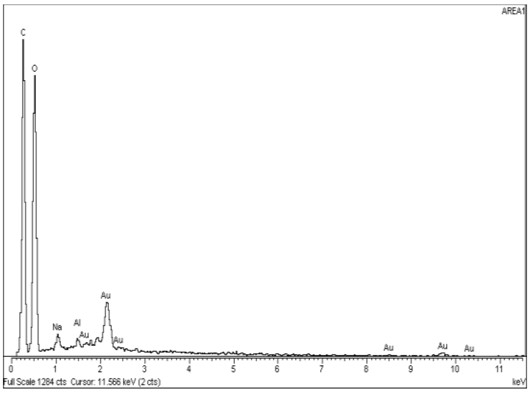

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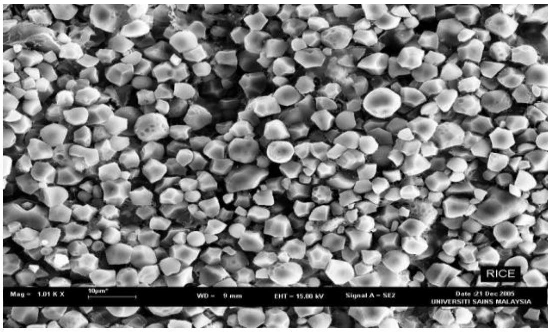
Fig.6. (a). SEM image of tapioca flour, (b). SEM image of filtered sludge using tapioca flour as a coagulant, (c). EDX analysis of sludge.

However, silicon is found absent in the EDX spectrum which could be due to the adsorption of rice starch granules surrounding them. Fig. 8(a) shows the SEM image of glutinous rice flour consisting of polygonal shapes and not in granular compound. This could be due to large volumes of waxy starches in glutinous rice flour. Fig. 8(b) shows the SEM image of the glutinous rice flour as sludge after the coagulation treatment.



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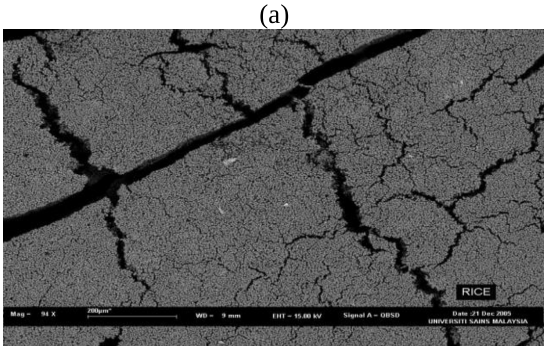

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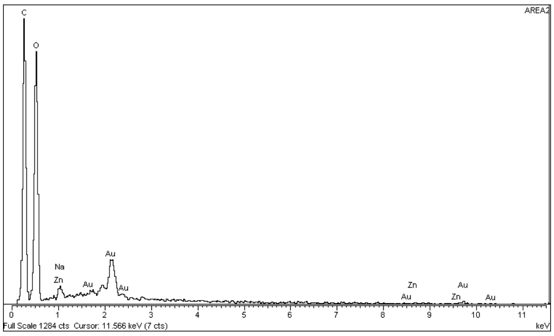
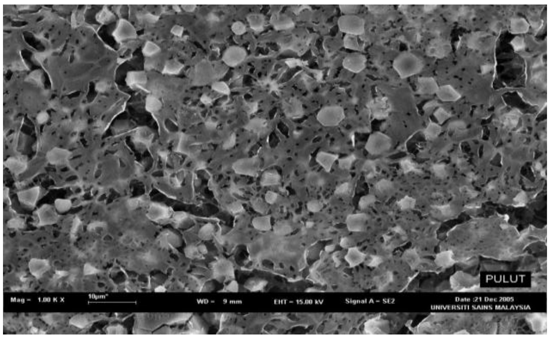


(c)

Fig. 7. (a). SEM image of rice flour, (b). SEM image of filtered sludge using rice flour as a coagulant, (c). EDX analysis of sludge.

The white spots on the sludge surface indicates that there are other elements present. From the EDX analysis in Fig. 8(c), we identified that the element was silica.

The present study showed that locally available flours i.e. corn, potato, tapioca, rice and glutinous rice could be used as natural coagulants to settle the silica content in semiconductor wastewater. From the SEM and EDX images analysis of the filtered sludge that were presented, it appeared that the natural coagulants employed in the study have similar characteristics with the commercial coagulants like PAC and alum on removing silica content from the semiconductor wastewater. This is interesting as our previous study were about the removal of COD and turbidity using sago and potato flour as natural coagulant agents. Concretely, our previous study showed that potato flour has the significant ability as a coagulant agent [20]. The mechanism of removing silica content from the wastewater is the polymer characteristic contained in the starch that traps the silica particles in the wastewater, causing them to be unstable and form large aggregates. From the SEM images, we found that the nanosize silica was adsorbed to the starches particles.



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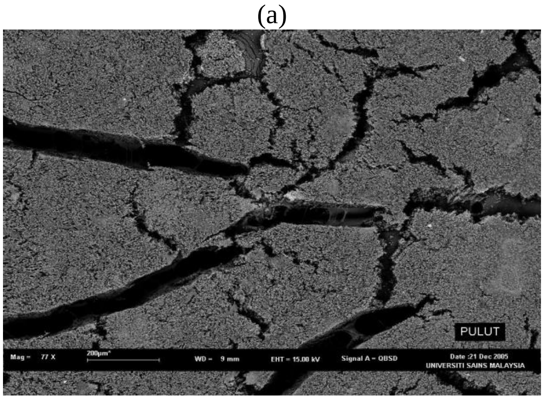

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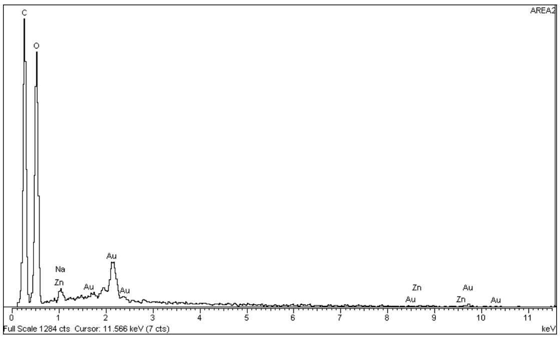


(c)

Fig. 8. (a). SEM image of glutinous rice flour, (b). SEM micrograph of filtered sludge using glutinous rice flour as a coagulant, (c). EDX analysis of sludge.

B. Removal of Heavy Metals Concentration

Table 2 presents the heavy metals concentration in the filtered supernatants which were analyzed after the coagulation treatment.

Table 1. Concentrations of heavy metal in the supernatant and raw semiconductor wastewater

Types of coagulant	Concentration (mg/L)		
	Zn	As	Cd
Tapioca flour	0.84	0.08	0.10
Corn flour	1.26	0.09	0.17
Glutinous rice flour	1.35	0.11	0.45
Potato flour	2.54	0.09	0.89
Rice flour	1.01	0.15	0.12
Alum	2.28	0.12	0.88
PAC	2.29	0.11	0.91

Three types of heavy metals were observed including arsenic, cadmium and zinc. Initially, these heavy metals were detected at trace levels in the raw semiconductor wastewater. We observed in comparison that the natural coagulants used in the study showed better metal adsorption performance than the commercial coagulants. Tapioca flour showed the highest metal adsorption, followed by corn flour and rice flour.

Settling the heavy metals concentration in semiconductor wastewater is strictly important. The presence of heavy metals in wastewater can pose significant health hazard to human and aquatic lives if the concentration exceeds the acceptable limits [19]. The standard limit of zinc, arsenic and cadmium concentration in Environmental Quality (Sewage and Industrial Effluents) Regulations 1979 set by the Environmental Quality Act 2006, Malaysia is presented in **Table 2**. We found that the commercial coagulants such as alum and PAC were not able to reduce the concentration levels of arsenic, zinc and cadmium in the supernatant. Conversely, tapioca flour was able to settle all three metal concentrations in the effluent. The other natural coagulants were also able to reduce the heavy metal concentrations below the standard limit, such as corn flour for zinc and arsenic, potato flour for arsenic, glutinous rice flour and rice flour for zinc.

Table 2. Legal requirement for heavy metal concentration in treated wastewater (source:

Environmental Quality Act, 2006, Malaysia).

Parameter	Standard limits (mgL^{-1})	
	A	B
Zn	2.00	2.00
Cd	0.05	0.10
As	0.05	0.10

C. Removal of Total Solids

Chemical coagulants such as PAC and alum were used to remove TS from the raw semiconductor wastewater. Results obtained are presented in **Fig. 9**. We observed that the TS concentration was proportionate to the increasing doses of the coagulants. The highest TS concentration was detected at 4367.91 mgL^{-1} using 0.02 to 0.03 gL^{-1} PAC (Fig. 9a), and 3058.82 mgL^{-1} using 0.02 to 0.06 gL^{-1} alum (Fig. 9b) at retention time of 60 minutes. The model description for the PAC and alum are as shown in (1) and (2):

$$Y_{PAC} = 4617.12 - 4.32X_1 + 177.93X_2 - 0.03X_1^2 - 32.27X_2^2 + 1.59X_1X_2 \quad (1)$$

$$Y_{alum} = 2568.13 - 14.73X_1 + 753.32X_2 + 0.28X_1^2 - 18.64X_2^2 - 1.58X_1X_2 \quad (2)$$

Fig. 10 shows the surface plot on the removal of TS content from the semiconductor wastewater using natural coagulants, such as corn flour (a), potato flour (b), tapioca flour (c), rice flour (d) and glutinous rice flour (e). We observed that the natural coagulants effectively removed the TS contents from the semiconductor wastewater. The TS of 2924.47 mgL^{-1} was achieved at the retention time range of 0 to 52 minutes using corn flour as a coagulant with the concentration range of 0.02 to 0.07 gL^{-1} (Fig. 10a). The highest amount of TS was 6626.67 mgL^{-1} after an addition of 3.8 to 6.0 mgL^{-1} of coagulant and retention time up to 15 minutes. The model description with the addition of corn flour as a natural coagulant is as shown in (3):

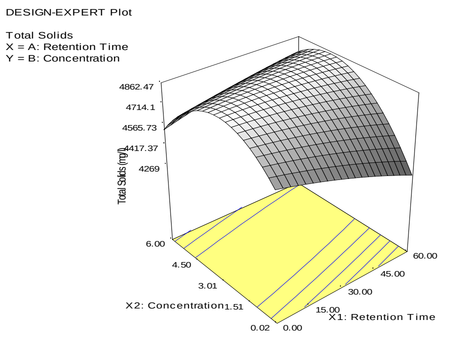
$$Y_{corn} = 2232.72 - 28.21X_1 + 1568.86X_2 + 0.76X_1^2 - 113.71X_2^2 - 7.38X_1X_2 \quad (3)$$

Fig. 10(b) shows the total amount of TS removal from the semiconductor wastewater using potato flour as a coagulant. About 2700 mgL^{-1} of TS concentration was detected in the coagulated sludge using 0.02 to 3.38 gL^{-1} potato flour at the retention time range of 49 to 60 minutes. However, we also observed that the TS is proportionate to the increase of potato flour concentration. The model description of the coagulation of semiconductor wastewater using potato flour as a natural coagulant is as shown in (4):

$$Y_{potato} = 5377.10 - 39.26X_1 - 76.34X_2 + 0.35X_1^2 + 58.60X_2^2 + 2.02X_1X_2 \quad (4)$$

Amount of TS removal of 2306.39 mgL^{-1} was obtained using tapioca flour as a coagulant at the retention time range of 42 to 60 minutes and at the coagulation concentration range of 0.02 to 3.01 gL^{-1} (Fig. 10c). However, the increase of TS concentration with increasing retention time and tapioca starch concentration was not proportionate. The model description using tapioca flour as a natural coagulant is as shown in (5):

$$Y_{tapioca} = 3249.65 + 0.42X_1 + 104.05X_2 - 0.55X_1^2 + 94.53X_2^2 + 1.07X_1X_2 \quad (5)$$



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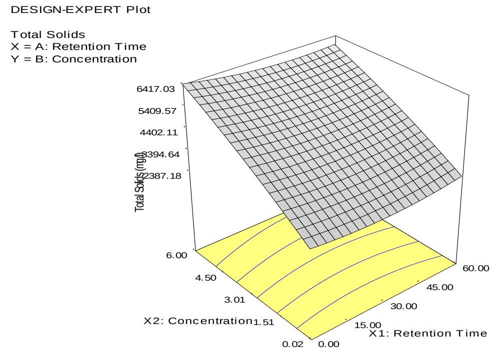

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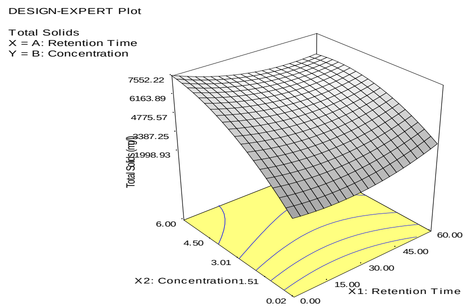
Fig.9. The effect of commercial coagulants on removal TS content from semiconductor CMP wastewater; (a) PAC, (b) Alum.

The TS removal was obtained about 1600 mgL^{-1} at the retention time of 30 minutes using 0.02 to 1.14 gL^{-1} rice flour as a coagulant (Fig. 10d). It was observed that with increasing doses of rice flour and at longer retention time increased the TS content in the sludge. However, the highest TS concentration was obtained about 5500 mgL^{-1} using 6 gL^{-1} of rice flour at the retention time of 60 minutes. The model description using rice flour as a coagulant is as shown in (6):

$$Y_{rice} = 611.25 + 54.26X_1 + 794.78X_2 - 0.72X_1^2 + 11.57X_2^2 - 1.71X_1X_2 \quad (6)$$

The TS removal of 3753 mg/L was obtained at the retention time range of 40 to 60 min using glutinous rice flour as a coagulant, as shown in Fig. 10(e). We observed that the TS was proportionate with the retention time. The model description for the TS removal from semiconductor wastewater using glutinous rice flour as a coagulant is as shown in (7):

$$Y_{gultinous\ rice} = 4151.03 - 13.58X_1 - 4.51X_2 + 0.24X_1^2 + 8.31X_2^2 - 2.36X_1X_2 \quad (7)$$



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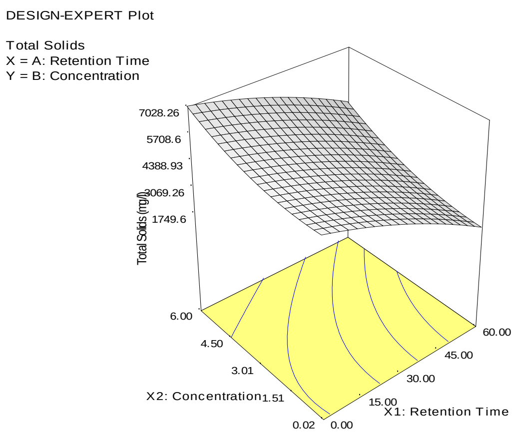

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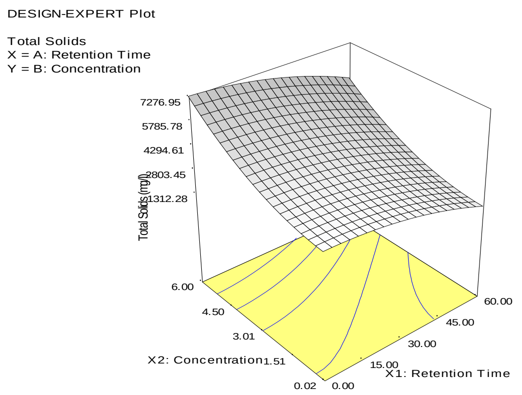

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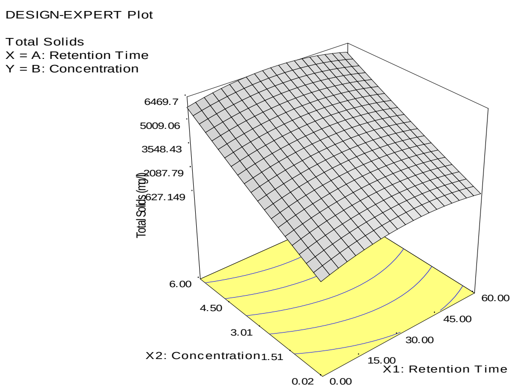

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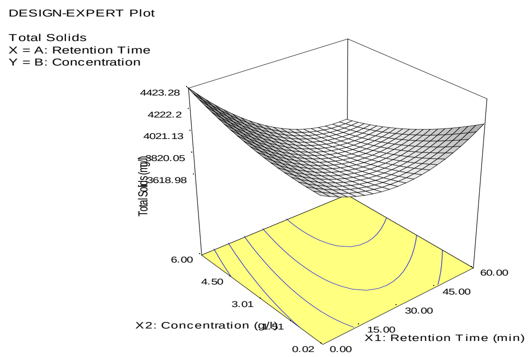

(e)

Fig. 10. Effect of locally available starches as natural coagulants on TS removal TS from semiconductor wastewater using corn flour (a) potato flour (b), tapioca flour (c), rice flour(d) and glutinous rice flour (e).

In the model equations, the positive sign in front of the terms indicates synergistic effect, whereas the negative sign indicates antagonistic effect. The quality of the model developed was evaluated based on the correlation coefficient value. The R^2 values were found to be 0.90, 0.89, 0.73, 0.77, 0.17, 0.53 and 0.78 for the coagulants of potato flour, tapioca flour, rice flour, corn flour, glutinous rice flour, PAC and alum, respectively. The R^2 values for potato flour, tapioca flour, rice flour, corn flour and alum which were obtained are relatively high, indicating that there was in good agreement between the experimental and the predicted values from the models. The highest TS removal were estimated by potato flour (90%), followed by tapioca flour (89%), alum (78%), corn flour (77%), rice flour (73%), PAC (53%) and glutinous rice flour (17%). Further, it can be estimated that the coagulation performance of the starches (except glutinous rice flour) on TS removal were almost similar to alum and higher than PAC. From the statistical results obtained, the above models were adequate to predict the removal efficiency of TS from semiconductor wastewater within the range of variables studied. Thus, the present study strongly reveals that the locally available starches have a bright future to be utilized as coagulants to treat semiconductor wastewater.

IV. CONCLUSION

The results of the present study show the feasibility of locally available starches as natural coagulants on the removal of total solids and reduction of heavy metals concentration in semiconductor wastewater. The SEM images of the coagulants and EDX analysis of coagulated sludge revealed that starches such as potato, tapioca, corn, rice and glutinous rice flour have similar coagulation characteristics with PAC and alum. Experimental results on the reduction of arsenic, cadmium and zinc concentration in the treated semiconductor wastewater showed that alum and PAC were not able to remove the metal concentration in the supernatant according to the standard limits set by the Environmental Quality Act, 2006, Malaysia. Conversely, the starches utilized in this study were able to decrease the metals concentration in the effluent. The factorial design and RSM analysis demonstrated that the experimental and the predicted values from the models were in agreement. The highest TS removal were estimated by potato flour (90%) followed by tapioca flour (89%), alum (78%), corn flour (77%), rice flour (73%), PAC (53%) and glutinous rice flour (17%).

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