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Application of Natural Starch Coagulant Followed by Membrane Filtration for the Elimination of Color From Stabilized Leachate

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ABSTRACT
The existence of humic substances is the key factor for antagonistic coloration of stabilized landfill leachate, which is a remarkable pollutant parameter. To examine the performance of natural starch from oil palm trunk (OPTS) like a coagulant is the focus of the current study, for the removal of color through coagulation-flocculation followed by membrane filtration. A series of jar tests were conducted for two different landfill sites through optimizing the dosage and pH followed by membrane filtration. The separation technique of starch from palm oil biomass, its morphology by scanning electron microscopy (SEM) as well as membrane filtration were discussed. Experimental results showed that the combination of coagulation-flocculation with OPTS as a novel natural coagulant and membrane filtration (CMF) yielded 81% and 82% of color removal for Pulau Burung and Alor Pongsu leachate respectively at optimum pH 6.0, a dosage of coagulant 1500 mg/L and 29° C temperature within a green approach than other conventional methods. FTIR spectrum peaks of leachate after CMF indicates the absence of certain groups of different wavelengths (wavelength 3100 cm^{-1}-3500 cm^{-1}, 1535 cm^{-1}, and 1900 cm^{-1} to 2100 cm^{-1}). The findings revealed that this CMF method can be a novel and sustainable alternative of harmful chemical coagulants for the removal of color from stabilized leachate.

Keywords: *Semi-aerobic landfill leachate, natural starch coagulant, humic substances, scanning electron microscopy*

1. INTRODUCTION

Municipal solid waste management is a rising and critical environmental issue in the context of rapid increment in population growth with economic development followed by an insufficient framework, proficiency, and land inadequacy. Because of landfill's cost-effectiveness & operational simplicity, it is still highly preferred as well as a suitable approach for solid waste management (MSW) apart from remaining the other options also. A complex hydrological and biogeochemical interaction occurs at the post dumping stage within landfills, which eventually causes the generation of a contaminated liquid recognized as landfill leachate [1]. This landfill leachate is an enormously poisonous complex wastewater stream containing large amounts of recalcitrant organic substances (humic & fulvic acid), ammonia nitrogen, heavy metals (lead, cadmium, chromium, copper), xenobiotic compounds, inorganic macro components such as manganese, calcium, magnesium, sodium, potassium, ammonium, iron, with high toxicity which raises destructive threats to the ecological system, food chain, and human wellbeing if discharged without necessary treatment [2].
Dissolved organic matter (DOM) is a diverse combination of refractory substances (humic and fulvic acids), proteins, carbohydrates, organic acids, and organic hydrocarbons that remain in matured landfill leachate. It is eminent that the existence of humic acid (about 60% of DOM), which is hardly biodegradable under normal conditions is mostly responsible for the greenish-black color of stabilized leachate [3]. To remove this color from matured landfill leachate is pretty complex and expensive because of the presence of a higher volume of refractory DOM, lower BOD_5/COD ratio [4], as well as higher ammonia[5].
Therefore, to implement appropriate landfill leachate treatment method becomes a major concern for the sustainability of environmental elements. To choose a suitable method for leachate treatment is contingent on focused parameters of leachate that essential to be removed. Among the commonly used methods, regarding humic acid removal with antagonistic color from stabilized leachate, advanced oxidation methods are not economically productive and responsible for chlorine oxidation, even though leachate recirculation is addressed one of the low-cost methods, yet it has the constraints of saturation as well as inhibitory effects of methanogenic activities, while ozonation, anaerobic filters, have also found nonproductive. Whereas, coagulation-flocculation method has been proven affordable and effective for the removal of high concentration refractory humic substances even with color & COD simultaneously [6]. Formation of flocs through destabilization of colloidal particles as well as charge neutralization of negatively charged colloids in the presence of natural or chemical coagulants followed by an amalgamation of impurities is the principle of this process [7].

Even though inorganic metallic coagulants are very well-known since 18th century [8] for leachate and wastewater treatment rather the sustainability of treatment process has several drawbacks since they are responsible for the neurological disease (Alzheimer) through penetration of the residual aluminium in the human body and brain [9], generation of excessive sludge volume, groundwater contamination by metallic toxicity [10], and so on. Whereas particularly natural coagulants from plant extracts are continuously accessible abundantly while recognized as toxic-free, renewable resources [11], and being environmentally affable, assumed safe for both terrestrial & aquatic ecosystem. Despite having some limitations due to satisfactory performance regarding pollutant removal nowadays natural starch-based coagulants (Hibiscus rosa-sinensis, chitosan, oil palm trunk, etc.) are being privileged over conventional chemical coagulants. Therefore, the implementation of natural coagulants to eliminate the refractory dissolved organic substances from stabilized leachate not so much well documented [12].

The natural starch consists of two major macromolecular polysaccharides, which are amylopectin as well as amylose. Besides, starch granule also contained other traces such as lipid, protein, ash, etc [13]. This study aims to explore the proficiency of natural starch-based coagulants separated from palm oil trunk as a substitute for conventional chemical coagulants exclusively for the removal of the color of stabilized leachate from two different sites, i.e. Pulau Burung and Alor Pongsu.

The extraction of natural starch from the biomass of palm oil to apply it as a coagulant treatment is extensively a new low-cost predecessor from agriculture waste. Malaysia is the second highest (about 35%) supplier of palm oil of the world and every 25 years it requires replantation. So, the accessibility of abundant biomasses is the key reason to focus on this agricultural product. The harvesting activity of oil palm trunk leaves behind about 80 million tons of residue every year undoubtedly, which creates a major disturbance to atmospheric chemistry, boost up the global climate change [14].

This study focused on oil palm trunk extracted starch (OPTS), an organic polymer with high molecular weight, to uphold how effective it is in the coagulation process. OPTS is a polyphenolic product remains in almost every part of the palm oil trunk (POT). Yin demonstrates [15], plant based polymeric coagulants are strongly conjoined with coagulation mechanisms through adsorption with charge neutralization by interparticle bridging. Figure 1 exposes the graphical illustration of elementary OPTS configuration with probable molecular correlations that stimulate the coagulation.

However, the ultimate outcomes of this study are anticipated to produce a comprehensive thought for the combined application of coagulation followed by membrane filtration (CMF) for treating the stabilized leachate. Complementary combination of coagulation and membrane filtration (CMF) appears to be a profitable, simple and competent approach for the exclusion of color.

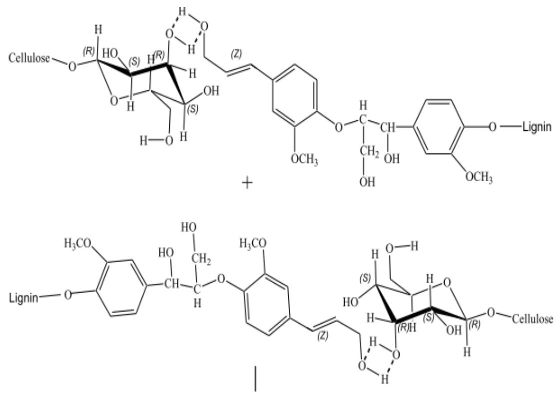


Figure 1 Bonding structure of oil palm trunk [16]

2. EXPERIMENTAL METHODS AND MATERIALS

2.1 Collection and Characterization of Leachate

The stabilized leachate samples were collected 6 times from the Pulau Burung Landfill Site (PBLS), and Alor Pongsu Landfill Site (APLS), Malaysia from August to December' 2019 avoiding the rainy days within 72 h before sampling using 20 L high quality plastic (polyethylene) cans with wrapped caps. The functional zone at Pulau Burung landfill site is about 35 ha with leachate pond and having a semi-aerobic system, which receives about 2,000 tons of solid waste daily. While, Alor Pongsu site is also termed as stabilized landfill operating from year 2000 and receives about 200 metric tons of solid waste per day. All samples were stored in laboratory cold room with 4°C temperature to maintain the purity of leachate.

Throughout the study, the leachate characterization were performed before and after each treatment, in terms of turbidity, pH, SS, color, Turbidity, COD, BOD_5, Dissolved Oxygen (DO), Total Dissolved Solids (TDS), zeta potential, and humic acid (HA) following the Standard Methods [17].

2.2 Extraction of Natural Starch from Palm Oil Trunk

The current study conducted the extraction of natural starch from Oil palm trunk biowaste (OPTS) through the novel bi-sulfite steeping method which was the modification of the DOS method demonstrated by Noor et al [18] to examine the performance as a natural coagulant. uniformly chipped OPT meal was steeped for 2 h in a 1:1.5 ratio under 0.5% sodium bisulfite solution after taking initial weight. The accumulation of sodium bisulfite solution crumbles the protein/starch matrices and restrains the growth of microorganisms. After 2 h of steeping and blending the OPT meal thoroughly, slurry was placed into a nylon screen for squeezing the slurry and placing the filtrates in a plastic dish. After sieving the filtrate with 212 µm sieve and 2 h settlement, the supernatant was removed by leaning the dish. Mixing 0.5% aqueous sodium bisulfite solution thoroughly (in 1:3 ratio) starch suspension was left for 1 hr settlement. The floated was discarded as earlier, ultra-pure water (1:0.33 ratio with the initial weight of POT) was added, centrifuged the mixture at 3,500 rpm up to 10 mins. The starch precipitates were filtered with vacuum filtration attired with a fiberglass filter of 1.6 µm opening to rinse as well as refine washed with 50 mL aliquots of acetone and finally, the starch was sun-dried for 24 h and crushed lightly to get fine powder- like starch.

Figure 2 Extracted Starch from oil palm trunk

2.3 Coagulation-flocculation and Membrane Filtration

Several batch experiments were carried out through coagulation for determining the removal (%) of color and optimizing the treatment condition regarding pH, coagulant dosage, etc. Modern jar-test device (Lovibond-Tintometer Group, Model ET 750, Germany) with impellers arranged by 25 mm × 75 mm rectangular blades, conducted the coagulation. Jar test followed three successive phases with 500 ml of well-conditioned leachate samples in each beaker and varying pH (4 to 7) as well as coagulant dosage (500 mg/L to 2500 mg/L): (a) rapid mixing phase with the speed of 150 rpm for 4 mins, (b) slow mixing phase maintaining variable speed and duration from rpm 15 to 25 and 10 to 30 mins respectively, & (c) allowing sedimentation time from 45 mins to 120 mins.

The microfiltration was applied using Hydrophobic Polyvinylidene (PVDF) membrane to remove humic acid (HA) from leachate after coagulation with novel modification of the previous method [19]. Leachate sample was filtered by 0.45 µm membrane filter and left for about 18 h with pH of 1.8 adjusted by 7(M) H_2SO_4. Then leachate was centrifuged at 4000 rpm up to 40 mins and then supernatant was removed from the precipitate, which is termed as humic acid (HA). Later this precipitate was dried overnight for 45˚C and grinded to get powder form of humic acid.

2.4 Analytical Method

All the chemical analyses were carried out by the standard methods for the investigation of water and wastewater [APHA 2012]. BOD_5 was measured according to the standard method 5210 B. Portable pH meter (Eutech Cyber Scan pH 510) measured the pH while turbidimeter (HACH 2100N determined the leachate turbidity. COD measurement was performed through the colorimetric process as per standard method 5220-D. TDS and DO were measured by digital YSI pro handled multimeter. Suspended solids (SS) and true color (465 nm wavelength) were measured using DR/2800 HACH spectrophotometer in unit of mg/L and platinum-cobalt (Pt-Co) respectively. The Malvern Zeta Sizer was used to measure the zeta potential and Malvern Master Sizer was used for flocs analysis. MS Excel®2016 software analyzed the results and indicate the parameter designs for optimization.

From the initial and final reading of color of leachate sample beneath 2 cm of surface., the removal percentage was calculated applying Eq. (1)

$$\% \text{ removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where, C_i = initial color reading of the leachate sample,

C_f = final color reading of the leachate sample

2.5 FTIR Spectroscopy

FTIR analysis was conducted for raw leachate and leachate after CMF using a Spectrum IR Tracer-100 Series FTIR (Shimadzu, Tokyo, Japan). At least three spectra were acquired for each sample. The spectra were in the range 4000 cm^{-1} to 400 cm^{-1} during the FTIR spectroscopy.

2.6 SEM Micrograph

The surface morphological analysis and energy-dispersive X-ray spectroscopy (EDX) were performed for the extracted starch from oil palm trunk using a SEM/EDX scanning microscope FEI-Quanta 450 FEG. A minor quantity of the starch was placed on a sample table applying conductive adhesive, then constrained plane and desiccated on a hot surface. For platinum coating (30 angstroms thickness) of the sample surface an ion sizzling device was applied.

Table 1 Characterization of raw leachate [20]

Parameters	PBLS			APLS			Standard[a]
	Minm.	Maxm.	Avg	Minm	Maxm	Avg	
pH	7.99	8.4	8.19	7.89	8.48	8.18	6.0-9.0
DO mg/l	0.28	1.95	1.11	0.09	2.89	1.49	…...
BOD_5 mg/l	90	243	166.5	105	276	190.5	…...
COD mg/l	3070	4142	3606	2952	5255	4103	400
BOD_5/COD	0.02	0.05	0.035	0.03	0.05	0.04	…...
Color (Pt-Co)	**4118**	**5960**	**5039**	**3967**	**5768**	**4867**	**100 ADMI**
EC (µs/cm)	19945	27489	23717	7245	22550	14897	…...
Turbidity (ntu)	190	295	242.5	105	319	212	…...
Zinc (Zn^{2+})	0.86	1.79	1.32	0.5	3.5	2	2
TDS mg/l	14534	16230	15382	1990	10255	6122	…...
Copper (Cu^{2+})	3.2	5.12	4.16	2.91	4.96	3.94	0.2
Iron (Fe^{3+})	4.1	4.89	4.49	4.05	4.97	4.51	5
Zeta Pot. (mV)	-28.9	-16.7	-22.8	-26.2	-18.4	-22.3	…....
TSS mg/l	236	970	603	363	847	605	50

a= Environmental Quality (Control of Pollution from Solid Waste Transfer Station and Landfill) Regulations 2009.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Stabilized Landfill leachate

Table 1 displays the details outcome of the physicochemical characterization of raw leachate from PBLS and APLS. The details outcome of the characterization of the leachate sample collected from PBLS and APLS are depicted in Table 1. The leachate from both sites is categorized as basic (maximum pH value 8.40 & 8.48 respectively), having concentrated color as well as COD reading of 3606 mg/l and 4103 mg/l correspondingly, which surpasses the 400 mg/l standard value (Environmental Quality Regulations 2009). The alteration of transitional organic acids into methane (CH4) and CO_2 during the methanogenic stage of the landfill decomposition, is responsible for high pH values. Moreover, a very low (<0.1) BOD_5/COD ratio of these leachate samples indicates greater stability, lower biodegradability, higher age of landfills with substantial contamination by organic refractory compounds [21]. BOD_5/COD proportion of young leachate remains up to 0.83 during the acidogenic phase while decreases to 0.1 at the matured stage during the methanogenic stage. The high concentration of suspended solids (603 mg/l & 605 mg/l respectively) and BOD_5 (max. 243 mg/l & 276 mg/l), which are significantly higher in comparison with the standard (20 mg/l) (Environmental Quality Regulations 2009), specified the existence of higher organic contents in the sample. The remaining dissolved organics (DOM) in stabilized leachate in the form of humic and fulvic acid are mostly responsible for the greater concentration of greenish-black color (5039 Pt-Co and 4867 Pt-Co on an average for PBLS and APLS respectively) of leachate. These dissolved organic substances are recalcitrant and the composition of humic and fulvic acid mainly, which are measured in COD. At normal pH, the average value of the zeta potential of both leachate samples was close to -23.0 mV.

3.2 Starch Characteristics

The yield of OPTS was calculated by measuring the extracted starch after the final stage concerning the initial weight of the palm oil trunk before blending. The starch yield is 13% from oil palm trunk which is remarkably higher than Noor and Mehta who described a less yield (7.15%) of OPTS. Because of phenolic discoloration, the starch color found light or wheat brown. The Z-average hydrodynamic size of the OPTS was approximately 7.15µm with bimodal size distribution like the previous study and as cereal starch wheat or barley. Applying polydispersity index (PDI), the broadness of starch molecular weight distribution was calculated additionally. The PDI value (0.719) of OPTS revealed its monodispersed nature and uniformity (<1).

3.3 Results of Coagulation followed by Membrane Filtration (CMF) for the Removal (%) of Color

3.3.1 Impact of Coagulant Dosage on Color Elimination

Figure 3 displays the impact OPTS dosage over color elimination during coagulation followed by membrane filtration. The results demonstrate that coagulation performance was extremely related to the OPTS dosage since the removal of color fluctuate within the variation of coagulant dose. The initial results of coagulation displayed that the OPTS is efficient identically at pH 6 [20]. Therefore, the best coagulant dosage for OPTS was determined by fixing the pH at 6. Maximum removal of color achieved 81% & 82% for PBLS & APLS correspondingly at optimum dose of 1500 mg/L.

3.3.2 Effect of Slow Mixing Speed and Time on Removal of Color

The impact of slow mixing speed and duration were tested too through current study, as these are also among the major influencing parameters to achieve the highest coagulation result. For attaining a satisfactory removal, slow mixing is essential to separate the coagulant adequately promoting particle smashes. Optimum speed (rpm) of slow mixing and duration is mandatory for the formation of enough large size particles for the maximum elimination in the settlement stage. Fig. 4 (a, b) show the impact of slow stirring speed and duration on color removal for both PBLS & APLS leachate. Results exhibited maximum removal of color achieved at 20 rpm and 15 mins mixing duration [22].

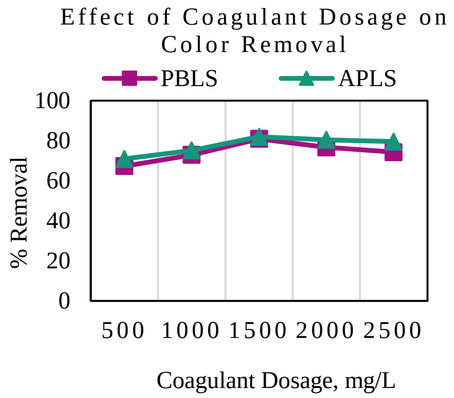


Figure 3 Effect of coagulant dose on color removal

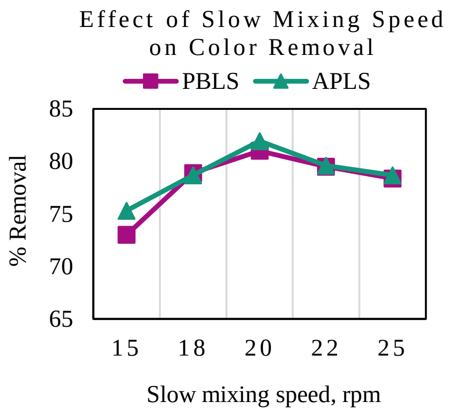


Figure 4 (a) Effect of slow mixing speed on color removal

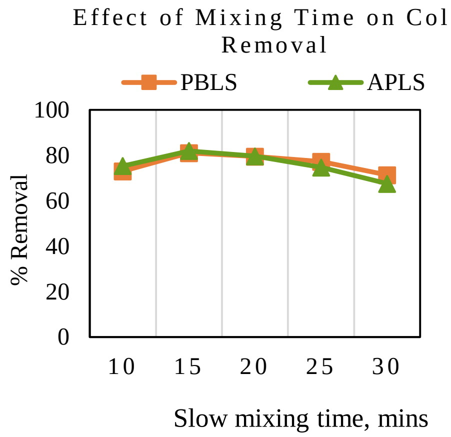


Figure 4 (b) Effect of slow mixing time on color removal

3.3.3 Consequence of Settling Period on Color Elimination

The impact of settlement time on color elimination is also remarkable for applying OPTS through coagulation (Fig. 5). Previous researches also revealed that [23]. The persistence of the settling process is to generate cleaner supernatant through dispersing the larger particles from leachate.
Experimental outcome indicates that the maximum color removal for both PBLS and APLS (according to t-test $p<0.05$) achieved when the sedimentation time reaches up to 90 mins.

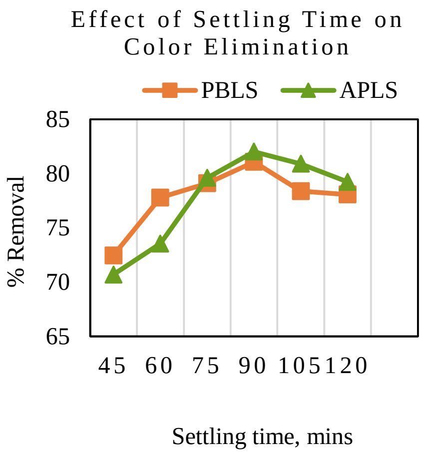


Figure 5 Effect of settling time on color elimination

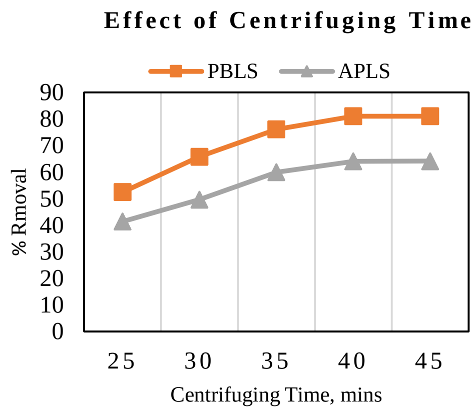


Figure 6 Effect of settling time on color elimination

3.3.4 Effect of Centrifuging Time on Removal of Color

This study investigated the effect of centrifuging time during membrane filtration at a constant speed of 4000 rpm. Fig. 6 shows the effect of centrifuging time on color removal over leachate collected from the PBLS and APLS site at the post membrane filtration stage after coagulation.

The achieved output of this investigation indicates that allowing optimum centrifuging time enhanced the removal of color satisfactorily. In this study, the optimum removal (according to t-test $p<0.05$) of color for both PBLS as well as APLS achieved when the centrifuging time reaches up to 40 mins.

3.3.5 SEM-EDX Investigations Output

Granular morphology of extracted OPTS were analysed through Scanning Electron Microscopy (SEM). SEM images (Fig 7) showed much greater granular sizes of OPTS. The outcomes were consistent with other OPTS granular structural analysis, featuring a mature storage of starch. The micrographs show that the granular architecture of OPTS is almost ovular and elliptical patterns with condensed ends. Bell-shaped granules were also observed. This outcome was almost similar with the explanations by Hashim *et al*. [16]. Figure 8 displays the outcomes of EDX result with the presence of several minerals i.e. Na, K, Mg, with carbon (c) and oxygen (o).

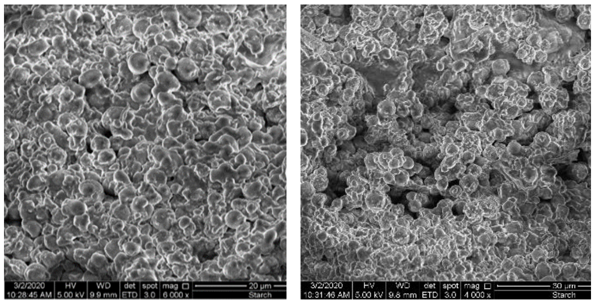
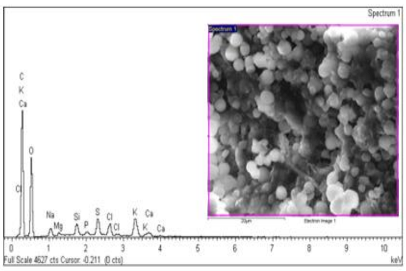


Figure 7 SEM images of starch particles in 4K and 6K magnification



Elements	Weight(%)	Atomic(%)
C K	55.83	64.62
O K	37.3	32.42
Na K	0.8	0.48
Mg K	0.21	0.12
Si K	0.77	0.38
P K	0.28	0.13
S K	1.38	0.6
Cl K	1.13	0.44
K K	1.89	0.67
Ca K	0.42	0.15
Totals	100	

Figure 8 EDX analysis outcomes of starch particles

3.3.6 Analysis of Flocs and Discussion

Figure 9 displays the images of flocs (9a) after coagulation at optimum treatment conditions and the humic acid portion after membrane filtration (9b). According to Figure 7 OPTS particles show a smooth surface with an almost uniform small unit, while the images of flocs displayed a further compacted, thicker with a larger structural view because of the bridging. According to Figure 7, particles are uniformly distributed with very clear and regular shape while Fig 9(a) displays irregular rough surface flocs with large size because of agglomeration. Moreover, based on the Mastersizer result, the difference in particle size of OPTS (7.15 µm) and sludge flocs (109 µm) is the evidence of the formation of the flocs.

The humic acid flocs became rougher, denser and larger than flocs after coagulation as displayed in Figure 9(b) because of the molecular accumulation within the flocs. The image indicated the gathering of the widespread black particles among the flocs, which in fact, expresses the idea of the molecular trapping within the floc accumulation. Within the acidic environment, nitrogen pair remains in amino groups changes to the positively-charged molecules, whereas the carboxyl group (–COOH) stays as bipolar form [24]. Meanwhile, higher pH or alkaline environment reduces the bridging flocculation through increasing repulsion among the particles.

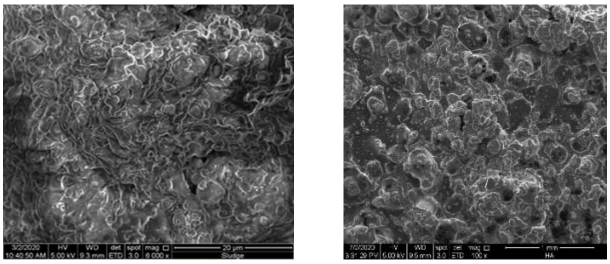


Figure 9 SEM images of (a) flocs after coagulation; (b) humic acid

Typically, coagulation performance is extremely subjected to the values of pH and zeta potential. Meanwhile, natural starch coagulants have limitations of charge stabilization in coagulation since they are negatively charged. To overcome this limitation chemical coagulants are preferred, which are responsible for the post- treatment severities over the environment.

So, prioritizing the environmental well-being this study investigated the removal efficiency of color by CMF without applying any traditional chemical coagulants.

Effective coagulation is the outcome of charge neutralization, floc formation, or bridging. Even though the surface charge of OPTS at normal pH (-18.1 mV) indicates particle instability, rather bridging is the vital mechanism of coagulation, especially for natural polymeric coagulants at comparatively lower pH. Inter-molecular bridging phenomena appear here by adsorbing starch on the molecular surface applying hydrogen bond. Higher molecular weight and a long polymeric chain is favourable for bridging with higher removal [25].

Significant impact of slow mixing speed (p-value = 0.00017) and its duration (p-value = 0.00051) specifies that coagulation displays better performance at 20 rpm with 15 mins duration. Figure 5 displays the impact of settling time, which is another substantial factor for the removal of color (p-value = 0.00068). Allowing optimum settling time enhances the opportunity of aggregating more flocs and settle subsequently. Considering all the experimental parameters throughout their investigated ranges regarding maximum removal (%) of color, the optimum condition for CMF will be as follows: coagulant dosage = 1500 mg/l, slow stirring speed = 20 rpm, duration= 15 mins, settlement duration = 90 min, centrifuging duration = 40 mins. At optimum condition, color removal for PBLS and APLS were obtained 81% and 82% respectively, which is much better than the removal efficiency demonstrated by early studies, who applied high dosage of chemical [26], or composite coagulants [27].

3.3.7 FTIR Analysis Results of Leachate

FTIR analysis was performed to study the variations of the organics functional groups of leachates after applying coagulation followed by the membrane filtration process. Four major peaks were frequently observed in the raw samples of leachate (Fig 10) at the wavenumber of 2081 cm^{-1}, 1636 cm^{-1}, 1444 cm^{-1}, and 1340 cm^{-1} represents several organic groups [28]. However, after CMF process mentionable changes in the spectrum were followed in leachate (Fig 10), including the absence of the 2081 cm^{-1}, 1444 cm^{-1} and 1340 cm^{-1} peaks, which are actually from refractory humic acid groups with highly decreasing trends of intensity for the peak at 1631cm^{-1}, indicating the degradation of organics due to CMF [29].

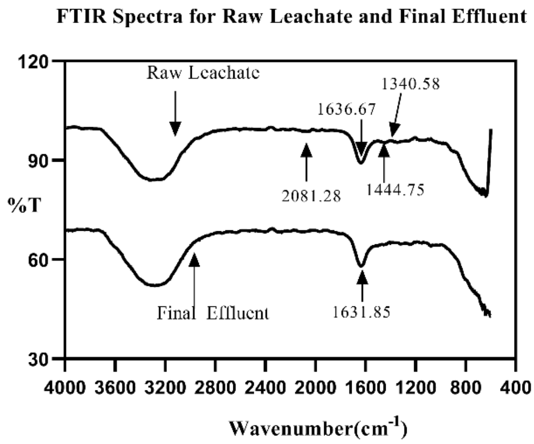


Figure 10 FTIR analysis results of raw leachate before CMF and final effluent after CMF

4. CONCLUSION

The coagulation followed by membrane filtration (CMF) process is proved to be a more efficient technique for the elimination of extreme color from semi-aerobic landfill leachate. Various operating parameters were studied to obtain the optimum treatment condition, which led to the maximum removal efficiency of color. The experimental output showed 81% and 82% color removal for PBLS and

APLS leachate at optimum coagulant dosage of 1500 mg/L, which are even better than the removal efficiency demonstrated by Zineb et al, who applied a high dosage of ferric chloride as a coagulant [26], Awang, who applied alum with hibiscus rosa- sinensis [27]. In a nutshell, this newly developed process could be considered as an advanced and sustainable technique for the elimination of color from matured leachate.

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