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Journal of Hazardous Materials 142 (2007) 477–482
Journal of Hazardous Materials
www.elsevier.com/locate/jhazmat

Innovative physico-chemical treatment of wastewater incorporating *Moringa oleifera* seed coagulant

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Received 18 May 2006; received in revised form 18 August 2006; accepted 21 August 2006
Available online 25 August 2006

Abstract

Moringa oleifera is a pan tropical, multipurpose tree whose seeds contain a high quality edible oil (up to 40% by weight) and water soluble proteins that act as effective coagulants for water and wastewater treatment. The use of this natural coagulant material has not yet realised its potential. A water extract of *M. oleifera* seed was applied to a wastewater treatment sequence comprising coagulation–flocculation–sedimentation–sand filtration. The study was laboratory based using an actual wastewater. Overall COD removals of 50% were achieved at both 50 and 100 mg/l *M. oleifera* doses. When 50 and 100 mg/l seed doses were applied in combination with 10 mg/l of alum, COD removal increased to 58 and 64%, respectively. The majority of COD removal occurred during the filtration process. In the tests incorporating alum, sludge generation and filter head loss increased by factors of 3 and 2, respectively. These encouraging treatment results indicate that this may be the first treatment application that can move to large scale adoption. The simple water extract may be obtained at minimal cost from the presscake residue remaining after oil extraction from the seed. The regulatory compliance issues of adopting 'new materials' for wastewater treatment are significantly less stringent than those applying to the production of potable water.

Keywords: *Moringa oleifera*; Wastewater treatment; Natural coagulant; Filtration; Alum; Coagulation

1. Introduction

Moringa oleifera Lam (*M. oleifera*) is a multipurpose tree native to Northern India that now grows widely throughout the tropics. A measure of the importance of the *M. oleifera* tree to communities across is the multitude of vernacular names ascribed to individual countries [1]. Many parts of the tree are used as traditional medicines, the seeds contain up to 40% by weight of quality edible oil (greater than 80% unsaturated fatty acid content [2]) and the seeds (and oil free presscake) yield proteins capable of acting as effective coagulants in water and wastewater treatment. The active components of the *M. oleifera* seeds have been determined to be cationic peptides of relatively low molecular weight (6–16 kDa) with an isoelectric pH value of 10 [3,4]. *M. oleifera* leaves are currently the focus of several development projects promoting their use as a valuable nutritional source for human consumption and to some extent as animal feed supplements [5].

In terms of water treatment applications, *M. oleifera* seed in diverse extracted and purified forms has proved to be effective at removing suspended material [6–13], generate reduced sludge volumes in comparison to alum [8,11], soften hard waters [14] and act as effective adsorber of cadmium [15]. These applications have been, and continue to be, fertile areas for researchers working primarily at laboratory level although some successful pilot and full scale water treatment trials have been reported [16,17]. However, this technology has not yet been adopted on any treatment plant at any scale: thus far, research activity has not led to sustained use. The major concern in the use of seed extracts for water treatment applications is the residual organic seed material that will be present in the finished water. Interactions with disinfectants such as chlorine and subsequent potential risks due to trihalomethane formation are possible. Residuals may also act as substrate for subsequent microbial growth. A recent study concluded that extraction in a salt solution followed by a single fractionation step (cation exchange

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doi:10.1016/j.jhazmat.2006.08.044

chromatography) is required to produce a protein rich solution for large volume treatment applications. Water and salt solution extracts are reported to yield protein mixtures of similar characteristics with the latter yielding approximately twice the protein yield of the former [4]. However, the technical and economic detail for scale up of the purification stage is not presented by the authors. Naturally derived coagulant materials must compete against proprietary coagulants (aluminium sulphate, ferric salts, polelectrolytes, etc.) on technical and economic terms: proprietary coagulants are sold in relatively high volumes at relatively low unit costs and the market is highly competitive and conservative.

In most countries, there are strict controls over the use of treatment chemicals such as those noted above. Previous individual studies indicate the non-toxic nature of *M. oleifera* seed in the context of water treatment. However, in order to obtain regulatory approval in any particular country, the safety of any proprietary seed product would have to be established by toxicological testing.

Wastewater treatment applications using crude water or salt extracts without further purification may represent the initial application for scale up. The use of chemicals in wastewater treatment is much less restricted by the regulatory agencies. This application could prove to be the necessary, initial developmental stage before large scale water treatment applications can proceed. This was the main objective of the work reported here: application of a water extract of *M. oleifera* seed to a wastewater treatment sequence comprising coagulation–flocculation–sedimentation–sand filtration.

1.1. Wastewater treatment considerations

Up to 70% of influent chemical oxygen demand (COD) in municipal wastewater is attributable to particulate matter larger than 0.45 μm [18]. Several pollutants are also incorporated into, or adsorbed onto, the particulate material. Thus, it is of interest to explore the development of a treatment strategy for the enhanced removal of suspended and colloidal solids from wastewater. Wastewater treatment is, to a very large extent, a matter of particle separation. This has led to the strategy of removing particulate and colloidal matter in the primary stage and thereafter dealing with soluble compounds that need to be transformed to colloidal matter and particulate matter (e.g. bacteria) before they are finally separated [19,20]. Wastewater treatment differs from water treatment in several ways. Water treatment is generally concerned with the removal of inorganic, hydrophobic colloids. Particulate matter in wastewater is mostly organic in nature and present in substantially greater concentrations; the average particle size is also greater. The mixing and flocculation conditions for the aggregation of organic colloids will be different to those employed for inorganic colloids: the hydrophilic surfaces of these organic particles react differently to coagulant addition [21].

If enhanced physico-chemical treatment at the initial wastewater treatment stage is effective, then the organic load on any subsequent biological treatment phase will be considerably reduced. The complete treatment plant will be more compact and more energy efficient [22]. Hence, chemically enhanced primary sedimentation (CEPS) has been advocated: inorganic salts and/or polymers are used as coagulant addition to enhance performance. It is postulated that non-settleable material carried over from the sedimentation stage may be amenable to removal by sand filtration.

The use of polymers as filter aids is one of the most effective methods to achieve the desired characteristics of influent particles to a filter [23]. When a polymer is used as a filter aid, interparticle bridging can increase the size of particles. If the polymer acts to neutralize the particle surface charge, the attachment can be enhanced by a reduction in electrical repulsive forces, and facilitation of mutual adsorption of a molecule or a group of molecules to both the filter media and particles. It has been reported that cationic polyelectrolytes can be used as filter aids to enhance the removal of suspended particles [24]. A patch mechanism can also take place due to the presence of low molecular weight and high charge density cationic polyelectrolyte [25].

The focus of this work was on particulate removal by settling and filtration. Any inorganic coagulant retained with suspended flocs will proceed and hinder the efficient operation of the subsequent biological treatment: biological treatment is essentially optimized to remove organic matter. Hence, it is felt that the application of *M. oleifera* as coagulant, which is organic matter and readily biodegradable, would be appropriate. If the particulates are removed, and the sludge generated proven to be non-hazardous by analysis, then the sludge may be used as a fertilizer and/or soil conditioner after stabilisation.

In the present study, *M. oleifera* alone, and in combination with alum, was applied to raw wastewater samples. A laboratory scale sand filter was used to remove suspended flocs carried over from the coagulated-settled wastewater.

2. Experimental methodology

2.1. Preparation of coagulant stock solutions

2.1.1. M. oleifera stock solution

Dry *M. oleifera* seeds were obtained from a commercial seed supplier. Mature seeds showing no signs of discoloration, softening or extreme desiccation were used [9]. The seed kernels were ground to a fine powder of approximate size 600 μm to achieve solubilization of active ingredients in the seed. Tap water was added to the powder to make 2% suspension (2 g of *M. oleifera* powder in 100 ml water). The suspension was vigorously shaken for 30 min using a magnetic stirrer to promote water extraction of the coagulant proteins and this was then passed through filter paper (Whatman No. 1). Fresh solutions were prepared daily and kept refrigerated to prevent any ageing effects (such as change in pH, viscosity and coagulation activity). Solutions were shaken vigorously before use [6,9,11].

2.1.2. Alum stock solution

The aluminum sulphate (alum) $[Al_2(SO_4)_3\cdot 18H_2O]$ used in this study was supplied by Bhandup water treatment plant, Mumbai, India. A 2% solution of alum in tap water was made (2 g of alum in 100 ml water). The alum was entirely soluble at this concentration.

2.2. Laboratory scale sand filter

A perspex transparent pipe of diameter 20 mm and length 750 mm was used as the filter housing for a 350 mm sand bed depth. Filter sand was also supplied by the Bhandup water treatment plant with grain size in the range 1.4–1.7 mm and uniformity coefficient 1.5. Plastic tubing of diameter 5 mm was used as piezometers inserted at 5, 10 and 25 cm bed depth as shown in Fig. 1. The supernatant from the coagulation–flocculation–sedimentation stage was passed through the sand filter using a peristaltic pump at a steady flow rate of 5 m/h. The filter was charged with fresh, clean sand prior to each filter run.

2.3. Experimental procedure

Wastewater samples were collected from Powai sewage pumping station, Mumbai, India. Typical wastewater characteristics and composition are given in Table 1. Beakers of 1 l capacity were used in standard jar test apparatus (Phipps and Bird Inc., USA). Pre-determined doses of coagulant were added while initially keeping the paddle rotational speed at 20 rpm. After the addition of the pre-determined doses of coagulant in all beakers, rapid mixing was conducted for 2 min at 100 rpm to disperse the coagulant. At the end of the rapid mixing period, slow mixing was continued at 20 rpm for 10 min to promote the flocculation of the suspended and colloidal particles present in the wastewater. After slow mixing, the beakers were carefully removed from the jar test set-up and the contents left to settle for 30 min. These jar testing criteria were established from a preliminary study. The supernatant from the settled wastewater was then filtered either through Whatman No. 1 filter paper (particle retention: 11 μm) or through the laboratory sand filter.

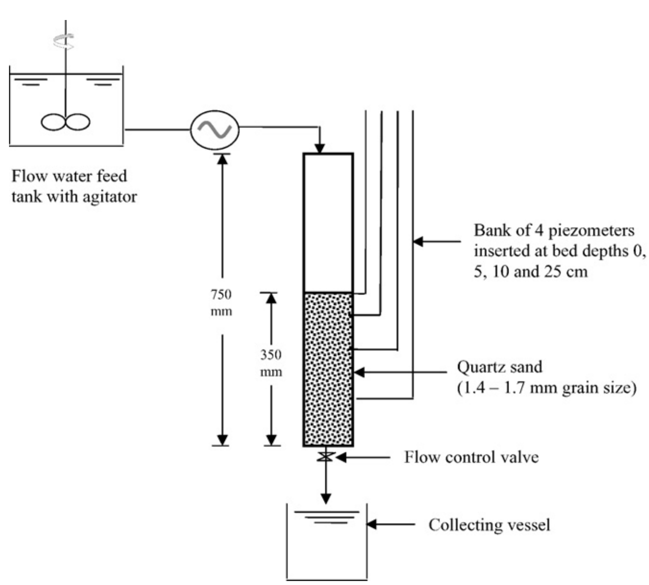

Fig. 1. Schematic diagram of laboratory scale sand filter.

Table 1
Indicative wastewater composition at Powai sewage pumping station

Parameter	Indicative value	Variation observed
pH	7.22	7.15–7.29
Turbidity	13.5 (NTU)	11–16 (NTU)
COD (mg/l)	150	140–224
Phosphorous (total) (mg/l)	3.06	2.25–3.88
Ammonia nitrogen (mg/l)	29.5	23.5–35.5
Nitrate nitrogen (mg/l)	1.34	0.18–2.5
Calcium (mg/l)	18.5	17–20
Calcium hardness (mg/l)	46.5	44–49
Magnesium (mg/l)	16.5	15–18
Total hardness (mg/l)	112.5	110–115
Alkalinity (mg/l)	215	215
Sodium (mg/l)	16.5	16–17
Potassium (mg/l)	10.5	10–11

Filter paper was used for the initial screening studies in order to ascertain the optimum dose of coagulant for a particular wastewater sample. Filter paper has been evaluated for use to simulate the actual performance of sand filters [26]. In another study, Whatman No. 1 paper (pore size: 11 μm) used for simulation produced a filtrate of less than 1 NTU [27]. In the present study, Whatman No. 1 filter paper was used for initial screening studies to establish the *M. oleifera* dose required for the actual sand filter runs. After evaluating the optimum dose, a sand filter run was conducted at the optimum dose. Turbidity, COD and head loss at different bed depths were measured at intervals throughout the filter run.

COD analyses were performed by the dichromate closed reflux method. Residual turbidities were used as a basis for comparing the efficiency of coagulation, which were measured by turbidity meter (Hach Chemical Limited, Model 2100 A, USA) in Nephelometric turbidity units (NTU). The procedures for COD as well as turbidity measurement conformed to those described in the Standard Methods for the Examination of Water and Wastewater [28].

3. Results and discussion

3.1. Sand filtration study with M. oleifera as coagulant

Fig. 2 gives the filtration characteristics with *M. oleifera* dosed at 50 mg/l. The initial COD value of 224 mg/l reduced to 196 mg/l after 30 min settling. It was this supernatant that was filtered at the flow rate of 5 m/h. The COD of the filtrate decreased at a relatively constant rate yielding a value of 112 mg/l (equivalent to 50% COD removal) after 150 min: the ripening period of the filter. The improvement in filter effluent quality results from particles captured in the bed serving as additional collectors for incoming particles. Between run time 150 and 300 min, the filtrate COD remained constant. Filter breakthrough of previously deposited particulates began at 300 min with both COD and turbidity increasing gradually from this time.

When wastewater passes through the porous filter, the flow experiences energy losses due to both form and drag

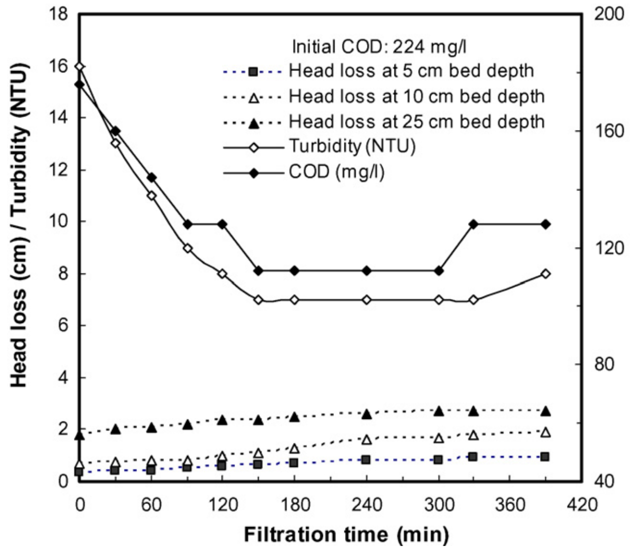

Fig. 2. Filtration characteristics of wastewater treated with 50 mg/l *M. oleifera*.

friction at the surface of the filter media material. Non-filterable flocs containing suspended solids and colloids settle on the sand grains creating head loss by further obstructing the passage of wastewater through the filter. After an initial period of 60 min, the rate of head loss development decreased marginally.

Fig. 3 gives the filtration characteristics with *M. oleifera* dosed at 100 mg/l. The initial COD of the wastewater was 192 mg/l and this reduced to 144 mg/l after settling. This supernatant was then passed through the filter at 5 m/h. The COD of the filtrate decreased at a relatively constant rate yielding a value of 96 mg/l (equivalent to 50% COD removal) after 120 min. This COD value remained sensibly constant at 96 mg/l until the termination of the run at 390 min.

3.2. Screening study with M. oleifera and alum coagulant addition

It has been reported by Cikurel et al. [29] that a relatively low alum dose of 10 mg/l alum yielded better wastewater filtration efficiency than higher doses: alum doses greater than 15 mg/l gave a bulkier, weaker floc. Tambo and Watanabe reported that alum floc density decreases with an increase in floc size [30]. Thus, in an attempt to explore improved filtration performance by the generation of a stronger, denser floc, 10 mg/l of alum was added in addition to the varying doses of *M. oleifera*. Fig. 4 gives the results obtained. With a combination of 10 mg/l of alum and 50 mg/l of *M. oleifera* (alum dosed first), the initial COD concentration of 144 mg/l reduced to 80 mg/l after settling. Thereafter, COD increased with increasing doses of *M. oleifera*. After filtration through filter paper (Whatman No. 1), there was a further significant decrease in COD to 45 mg/l with the 10/50 mg/l dosing combination. This represents total COD removal of 69%. Encouraged by this result, sand filter runs were conducted at 50 mg/l and 100 mg/l *M. oleifera* doses combined with 10 mg/l of alum.

3.3. Sand filtration with M. oleifera and alum coagulant addition

Fig. 5 gives the filtration characteristics with *M. oleifera* dosed at 50 mg/l together with alum at 10 mg/l. The initial COD of 192 mg/l reduced to 144 mg/l after settling. As before, the supernatant wastewater was passed through the sand filter at 5 m/h. Filtrate COD decreased over the initial 120 min, leveling at a value of 80 mg/l (equivalent to 58% COD removal). This value remained constant until rising at 330 min into the run.

Fig. 6 gives the filtration characteristics with *M. oleifera* dosed at 100 mg/l together with alum at 10 mg/l at the standard filtration rate of 5 m/h. The initial COD of 224 mg/l reduced to

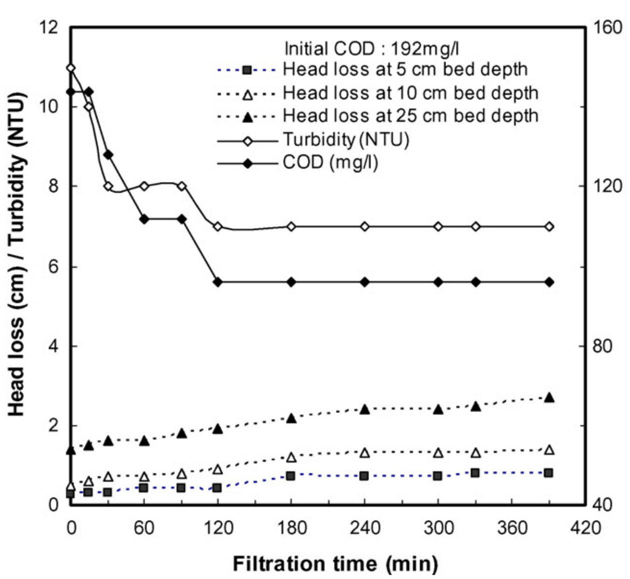

Fig. 3. Filtration characteristics of wastewater treated with 100 mg/l *M. oleifera*.

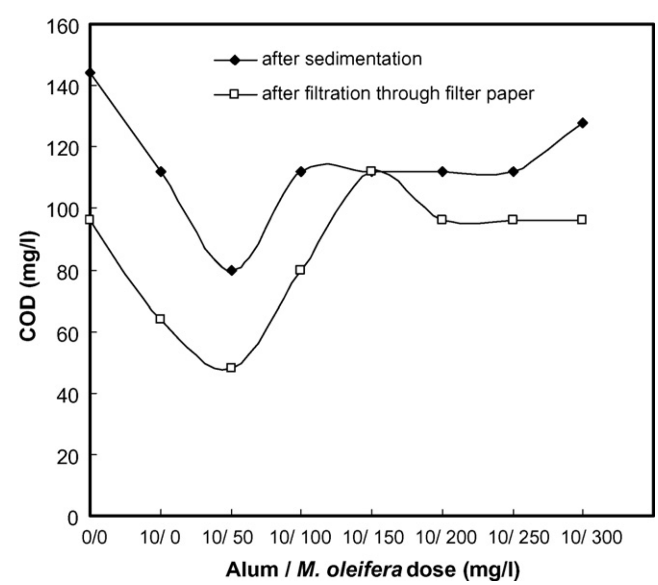

Fig. 4. COD dose response: alum and *M. oleifera* in combination.

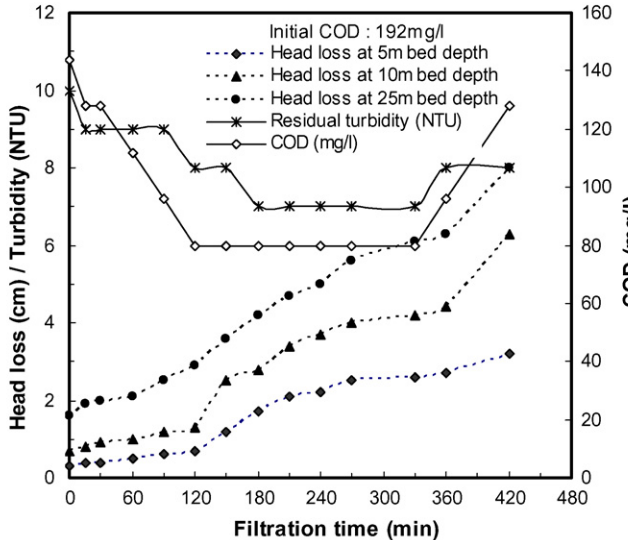

Fig. 5. Filtration characteristics of wastewater treated with 10 mg/l alum and 50 mg/l *M. oleifera*.

176 mg/l after settling. The filtrate COD decreased over the initial 120 min, leveling at a value of 80 mg/l (equivalent to 64% COD removal). This value remained constant throughout the filter run of 360 min.

3.4. Observations from treatment studies

Overall COD removals of 50% were achieved at both 50 and 100 mg/l *M. oleifera* doses. When 50 and 100 mg/l *M. oleifera* doses were supplemented by 10 mg/l of alum, further COD removals increased to 58 and 64%, respectively. At 50 mg/l of *M. oleifera* applied, either alone or in combination with 10 mg/l alum, COD breakthrough was evident at 330 min into the filter run. When 100 mg/l of *M. oleifera* was applied, either alone or in combination with 10 mg/l alum, COD breakthrough was not observed over the filtration run times. This breakthrough lag

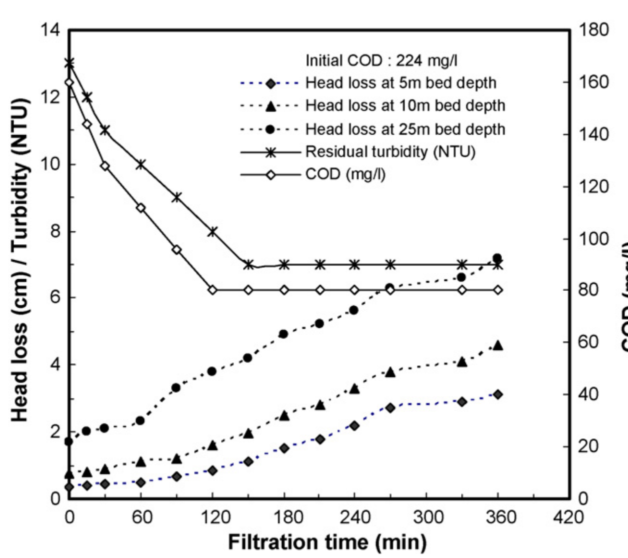

Fig. 6. Filtration characteristics of wastewater treated with 10 mg/l alum and 100 mg/l *M. oleifera*.

Table 2
Sludge volumes for different coagulant doses and combinations

M. oleifera dose (mg/l)	Alum dose (mg/l)	Sludge generated (ml/l wastewater)
50	–	3.0
100	–	3.0
50	10	6.5
100	10	6.0
–	10	8.5

time period (not quantifiable in these filter runs as breakthrough was not reached) may be due to relatively stronger flocs formed at the relatively higher *M. oleifera* dose: the stronger flocs being more likely to resist the hydraulic shear forces encountered during filtration. The majority of COD removal occurred during the filtration process. This removal may also be attributed to the relatively strong *M. oleifera* flocs that formed: being non-settleable but filterable [13]. The filter ripening period was relatively long for most filter runs. The filter head loss increased by a factor in excess of 3 when alum was dosed in addition to the *M. oleifera* coagulant. Low molecular weight cationic polymers have been observed to develop less head loss [23]. As previously noted, the active proteins of *M. oleifera* are as such. Measured sludge volumes generated with alum dosed alone is almost three times that of using *M. oleifera* alone (Table 2). This is in agreement with other studies that have reported *M. oleifera* sludge production being significantly less (by a factor of up to 5) than that produced by alum [12]. In this study, sludge produced while using the combination of *M. oleifera* and alum was approximately half that of alum when used as the sole coagulant.

4. Conclusions

In the wastewater treatment sequence investigated *viz.* coagulation–flocculation–sedimentation–filtration, the maximum overall removal of 64% of COD was obtained with the coagulant combination of 100 mg/l of *M. oleifera* with 10 mg/l alum. The majority of removal is evident during the filtration stage. These are encouraging results. The organic loading on any subsequent biological treatment stage would be much reduced. Future work is planned to further explore this area by undertaking laboratory study of direct and dual filtration of wastewater as an effective primary treatment step before biological stabilisation.

There are issues to address and overcome before large-scale commercialisation is possible, including: standardisation of the products, securing compliance with relevant regulatory approvals (such as toxicological studies) and development of markets (local/regional/international). In-country processing will add a real value and stability to the products and their markets. This represents an exciting initiative and challenge for many countries. Multidisciplinary collaboration and rigorous economic analyses are essential elements for future and continued success [16].

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