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Usage of UASB Reactor to Assess Feasibility of Treatment of Paper Mill Effluent

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Upflow anaerobic sludge blanket (UASB) reactors R-I and R-II, each with an effective volume of 6.0 l were used to study the treatability of actual effluent obtained from paper mills at a mesophilic temperature and neutral pH. Methanol, as a source of an easily biodegradable substance along with activated carbon of effective size 1.5 mm–2.5 mm were added to the reactor R-I to a total depth of 12 cm to evaluate its efficiency. The pH of both the reactors were kept constant at neutral by adding an external buffer solution of 0.03 M $NaHCO_3$ with the feed solution. It was observed that corresponding to an organic loading rate of 3.5 kg-COD/m^3-day, the overall chemical oxygen demand (COD) removal efficiency of the reactors R-I and R-II were 88% and 64%, respectively. The absorbable organic halides removal efficiency was observed to be 72% and 47% for reactor R-I and R-II, respectively. During the study it was however observed that, the treatability efficiency of reactor R-I was comparatively better but the amount of its biogas production was slightly lower than that of R-II. The average biogas production in reactors R-I and R-II during the course of study was observed as 0.33 l/g-$COD_{removed}$ and 0.42 l/g-$COD_{removed}$ respectively, with a mean methane composition of 58%–61% in both the reactors. Kinetic coefficients of k, K_s, Y and k_d were determined to be 0.7 g-TOC/g-VSS.d, 0.30 g-TOC/l, 0.26 g-VSS/g-TOC and 0.02 day^{-1} respectively, based on the results obtained from reactor R-I. The results of this study showed that the use of methanol and an activated carbon in a UASB reactor to anaerobically digest the paper mills effluent at a mesophilic temperature and a neutral pH reactor was quite a feasible and viable technique.

Key words: anaerobic digestion; activated carbon; methanol; COD; AOX; organic loading rate; design; parameters, pulping process; removal efficiency

Developing nations are highly subject to extensive environmental pollution, mainly due to the discharge of untreated industrial and domestic effluent. The paper mill industry is considered to be among the top-most pollution sources and releases a variety of toxic and hazardous wastes. It creates a massive threat to the environment by generating highly polluted effluent. The bleaching section of the mill discharges the most toxic type of effluent containing AOX (absorbable organic halides), which are produced as a result of the chemical combination of chlorine that comes from the bleaching section with the residual lignin of the pulping effluent (Ali *et al*. 2001; Savant *et al*. 2005). AOX are highly hazardous because the majority of members of this family are bioaccumulative, persistent and carcinogenic in character. Dioxin, which is recognized to be the most deadly substance ever found on earth, also belongs to the same family of AOX (Arshad *et al*. 2009).

A variety of physical, chemical and biological techniques have been attempted to decrease the concentration of AOX in paper

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mill effluent. As the physical and chemical processes are extremely costly in terms of their operational and maintenance costings, biological treatment processes are generally chosen. Among the biological techniques, the anaerobic technologies are regarded as more feasible options, particularly for developing countries because of their minimal amount of energy and nutrient consumption (Bhatti *et al*. 1996). Moreover, the implementation of anaerobic technology has been demonstrated to be comparatively easy and cheaper while it can also be used for a variety of industrial and domestic waste treatments including the effluent of paper mills (Lettinga *et al*. 1980).

At present numerous anaerobic treatment systems are functioning effectively around the world for the treatment of paper mill effluents (Bajpai 2000). Although they are extremely acknowledged for their treatability performance (Schellinkhout 1993), only insufficient work has been reported so far regarding the use of UASB reactors to eliminate AOX from the effluent of paper mills using NSSC (neutral sulphide semi-chemical) pulping process, which is the common pulping technique being practiced in Pakistan (ETPI Pak-EPA 1999). It was earlier noticed that anaerobic technologies were able to reduce 40%–60% of the AOX concentration (Fitzsimons *et al*. 1990; Ferguson & Dalentoft 1991). However, it has now been demonstrated that about 96% of the phenolic compounds can be removed at a hydraulic retention time (HRT) of 30 h (Anushuya & Gupta 2008; Rajakumar & Meenambul 2008; Mini Bajaj *et al*. 2009) but it's uneconomical to practically design a new UASB reactor merely on the basis of a longer retention time. Through its efficiency for the treatment of other kind of wastes like textile mill effluent, its COD removal has been reported to be more than 90% at a quite relatively reasonable HRT (Wijetunga *et al*. 2008)

It has been observed that the treatment efficiency of a UASB reactor could be enhanced by adding an additional source of an easily biodegradable substance to the reactor (Scholz *et al*. 1995; Bajaj *et al*. 2009). Adding methanol, the removal efficiency of the chlorophenolic wastes (members of the AOX family) in a UASB reactor had been observed to improve (Arshad & Hashim 2008) and also using an activated carbon with the digested sludge in an UASB reactor increases its performance (Mahadevaswamy *et al*. 2004; Hiroshi & Masasumi 2009; Arshad & Hashim 2010). Therefore, this study was specifically designed to determine the optimum design parameters i.e. organic loading rate (OLR) and hydraulic retention time (HRT) for the treatment of paper mill effluent employing the NSSC pulping process under anaerobic conditions. The main objective of this study was to examine the removal efficiency of UASB in terms of COD and AOX reduction using actual paper mill effluent in the presence of methanol and an activated carbon.

MATERIAL AND METHODOLOGY

UASB Reactors Specification

The UASB reactors (namely R-I and R-II) used were made of acryl resin material, each with an effective volume of 6.0 l. Water jackets were provided around the reactors to maintain a constant mesophilic temperature. A mixing device (turbine shape, 3.81 cm × 7.62 cm) and a gas separator system were also provided in both the reactors (Arshad & Hashim 2008). A systematic diagram of the UASB reactor is shown in the Figure 1.

The UASB reactor R-I was filled with granular activated carbon (effective size 1.5 mm–2.5 mm) to the total depth of 12.7 cm (Arshad & Hashim 2010).

Wastewater Characteristics

Actual wastewater sludge used in the study was obtained from the nearby local paper mill

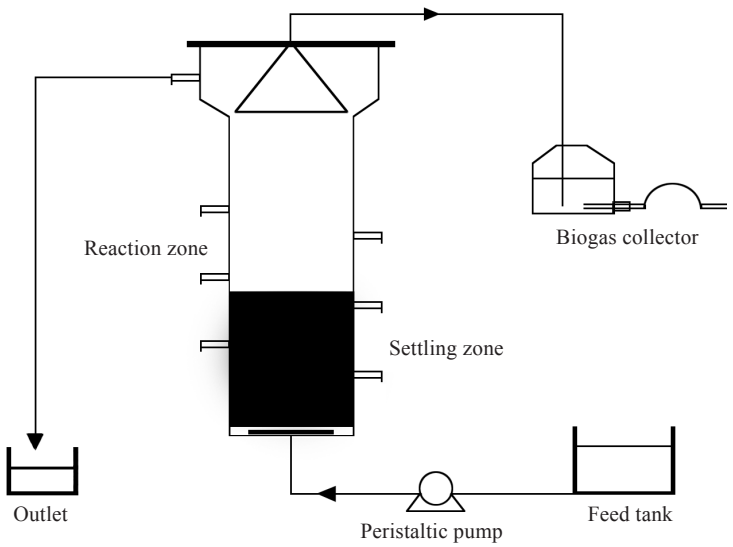

Figure 1. Systematic diagram of UASB reactor.

using NSSC pulping process. The wastewater characteristics data of the paper mills effluent is shown in the Table 1.

Substrate and Nutrients

Methanol was added to the feeding solution in reactor R-I to equalize its COD concentration to that of the actual effluent, by first diluting the same effluent with tap water. The ratio of methanol to actual effluent in the feeding solution to reactor R-I was kept around 1:5 (Arshad *et al*. 2010) during the course of study. For reactor R-II, the actual effluent without methanol was used as a feed solution.

Nitrogen and phosphorous were added to the feeding solutions of both the reactors, R-I and R-II in the form of $(NH_4)SO_4$ and KH_2PO_4, respectively, in accordance with the C:N:P ratio of 300:1:0.1 (Athar *et al*. 2008). $MgSO_4.7H_2O$ was also added to both the reactors at the concentration of 0.1 g/l (Bhatti *et al*. 1996).

Seeded Sludge

A digested sludge fully acclimatized with paper mill effluent in the laboratory for about three weeks was added to both the reactors for the start-up (Yoochatchaval *et al*. 2008). The concentration of MLSS and VSS of the seeded

Table 1. Wastewater characteristics of the paper mill effluent.

Parameters	Concentration
Ph	8.2
Colour (units)	1650
COD (mg/l)	2984
AOX (mg/l)	22.08
Total solids (mg/l)	4562
Total volatile solids (mg/l)	1774
Total dissolved solids (mg/l)	2386
Total suspended solids (mg/l)	1094

sludge were observed to be 62.28 g/l and 54.55 g/l, respectively.

Experimental Analysis

The pH, temperature, COD, AOX etc of the influent and effluent of the reactors were analyzed and recorded regularly twice a week. Total gas production was monitored using standard NaCl solution. All types of analysis were carried out using standard laboratory techniques (AWWA 2005).

RESULT AND DISCUSSION

Operating Parameters

Both the reactors were started up simultaneously and parallel according to the typical guidelines (Lettinga *et al*. 1984). As the pH and temperature were the two most important and principle operational parameters of anaerobic digestion, great care was therefore taken towards their control during the course of the study period. Neutral pH was considered to be the most suitable range for microbial activities during anaerobic digestion (Bhatti *et al*. 1996), thus the pH of both the reactors were maintained around neutral by adding an external buffer solution in the form of 0.03 M $NaHCO_3$ to the feed solution after a few days of operation when a drastic drop of pH was observed in these reactors. The time course of pH during the study period of the reactors is shown in Figure 2.

The initial pH of both the reactors was slightly above neutral at the start of the study but later on a decreasing trend was observed after the second week. The pH of the reactors R-I and R-II suddenly dropped down to 5.51 and 5.02, respectively. This normally happens under anaerobic conditions due to the accumulation of excess volatile fatty acids within the system during the start-up period but it can be controlled by adding an external buffer solution to the feed solution of the reactors. In this study, 0.03 M $NaHCO_3$ was used as the source of an external buffer to control the pH around neutral (Bhatti *et al*. 1996; Mtethiwa 2008). The average pH of both the reactors R-I and R-II after the addition of an external buffering solution was observed to be 7.10 and 7.22, respectively during the course of study period, which also validated the effectiveness of using an external buffer solution (Arshad *et al*. 2010).

Similarly, for anaerobic digestion a mesophilic range of temperature was considered to be an optimum and suitable range (Henze *et al*. 1983) because at lower temperatures (Psychrophilic range) the microbial activity

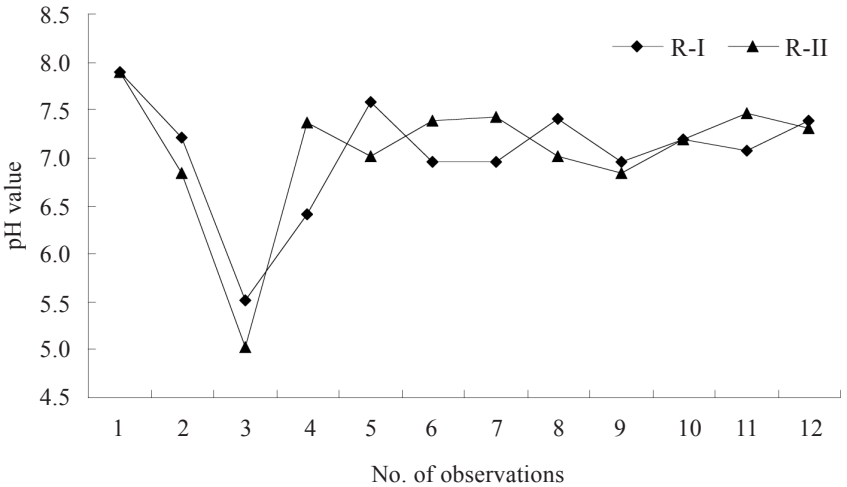

Figure 2. Time course of pH during the study period.

becomes quite slow (Switzenbaum *et al*. 1980; Kennedy *et al*. 1982; Grin *et al*. 1985) while at higher temperatures (thermophilic range) a number of potential problems arise, e.g. high endogenous death rate (Henze *et al*. 1983; Buhr *et al*. 1977). Therefore, in this study the temperature of both the reactors was kept constant at 27°C–33°C by using external heating devices (water jackets).

Both the reactors R-I and R-II were started up simultaneously and in order to avoid organic shock to the reactors, the OLR was gradually increased starting from 0.2 kg-COD/m^3-day to 5.5 kg-COD/m^3-day and the hydraulic retention time (HRT) was slowly decreased from 78 h to 6 h during the course of the study period.

Treatability Evaluation of the Reactors

OLR and HRT are the important design parameters of wastewater treatment systems which determine the capital cost and establish their engineering and economic feasibility. During this study, the effects of OLR and HRT on the removal efficiency of COD and AOX concentrations in the reactors were observed thoroughly and the relevant data obtained during the course of the study period has been plotted as shown in Figures 3–6.

The data indicates that the treatment efficiency of the reactors R-I and R-II was greatly influenced by the OLR and HRT. The lower OLR and higher HRT seem to be in quite favourable conditions for achieving higher treatment performance under anaerobic conditions in both the reactors R-I and R-II in terms of COD and AOX removal, and vice-versa. Such operational conditions of lower OLR and higher HRT make the system design uneconomical when applied to larger field scales. It was noticed that for every increase in OLR or decrease in the HRT, there was an abrupt decrease in the treatability performance of reactors R-I and R-II. This might be due to the sudden shock of a heavy organic load or the excessive accumulation of organic acids within the systems.

The data indicated that corresponding to optimum operating conditions, i.e. OLR of 3.5 kg-COD/m^3-day and HRT of 22 h, the overall COD removal in reactors R-I and R-II was 88% and 64%, respectively. The AOX removal in reactors R-I and R-II corresponding to same operating conditions were noticed to be 72% and 47%, respectively. Throughout the study, it was observed that the reactor R-I (with activated carbon) gave comparatively better treatment efficiency than the reactor R-II

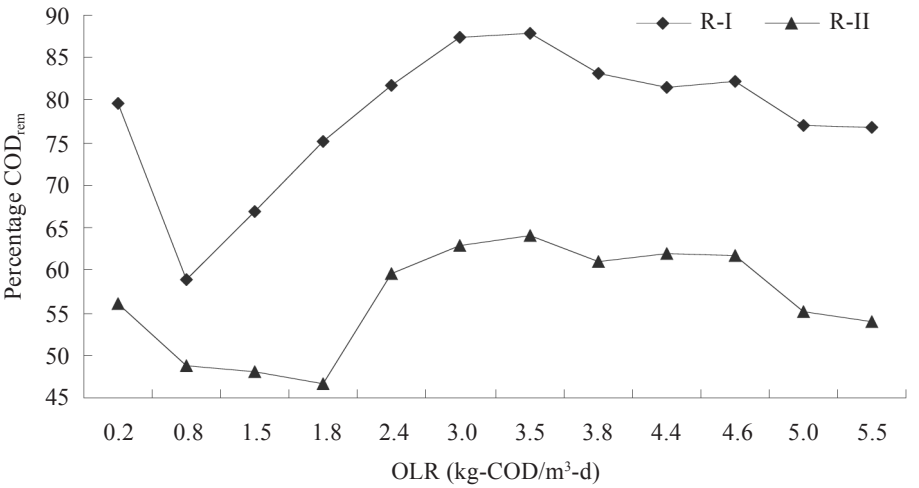

Figure 3. Effects of OLR on COD removal.

(without activated carbon), which indicated that the addition of activated carbon to the UASB reactor enhanced its treatability performance.

The previous studies showed that anaerobic digestion, in combination with other treatment systems like aerobic, membrane filtration etc. is able to remove 40%–65% AOX (Lee *et al*. 1993; Hall *et al*. 1995; Francis *et al*. 1997; Tezel *et al*. 2001) but if it is used single-handedly than only 42%–45% of AOX could be removed (Fitzsimons *et al*. 1990). Ferguson and Dalentoft (1991) reported 40%–65% AOX removal while observing the treatability efficiency of the bleaching effluent of pulp and paper mills under anaerobic conditions. Comparison between COD and AOX removal efficiencies of the UASB reactors based on similar studies is mentioned in the Table 2. The present study seemed to be more reliable and gave better treatment efficiency of COD and AOX. This was because the new strategy was modified to work under anaerobic conditions in an UASB reactor where methanol as the source of an easily biodegradable substance was added to the feeding solution and also the usage of

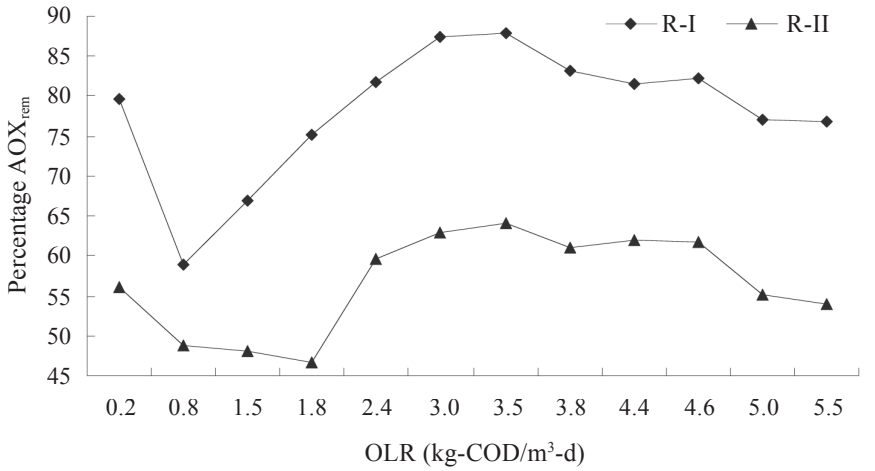

Figure 4. Effects of OLR on AOX removal.

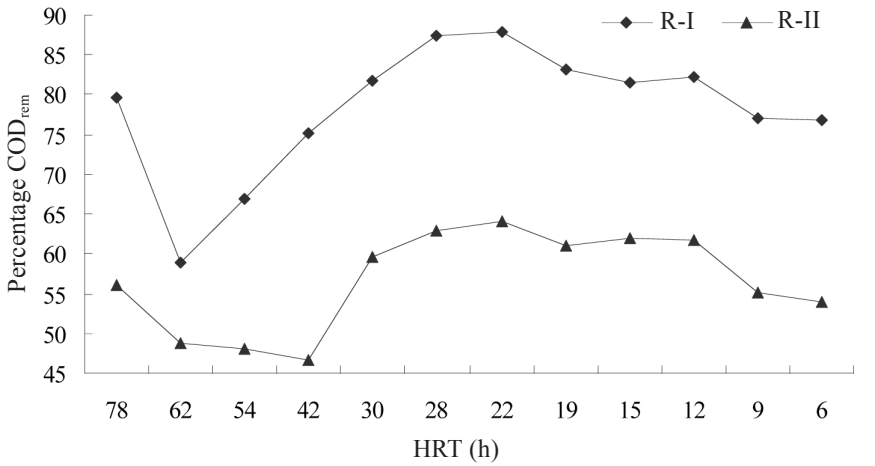

Figure 5. Effects of HRT on COD removal.

activated carbon with the digested sludge. The Figures 4–6 provide practical design aid for the UASB reactor to treat paper mill effluent employing the NSSC pulping process.

Production of Biogas

Initially small gas bubbles were observed during the starting period. Appropriate collection of the biogas was done at the end of second week. The gas collection system consisting of saturated NaCl solution was installed in the reactors R-I and R-II. The data obtained pertaining to the biogas generated during the course of the study period is illustrated in Figure 7.

It was observed that the two design parameters i.e. OLR and HRT had important roles on the amount of biogas production under normal conditions of pH and temperature. The amount of biogas generated is directly related to the amount of feeding solution in the reactor, and the HRT plays a significant role in controlling the rate of biogas production. It was observed that there was a significant reduction in the amount of biogas production for both the

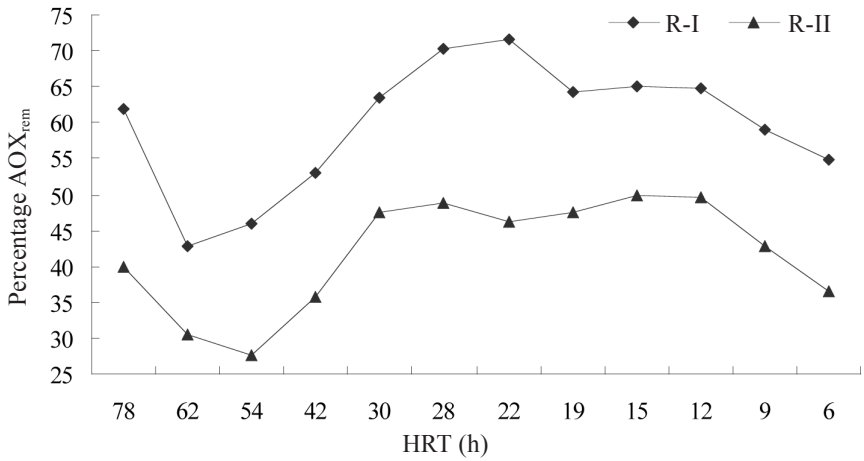

Figure 6. Effects of HRT on AOX removal.

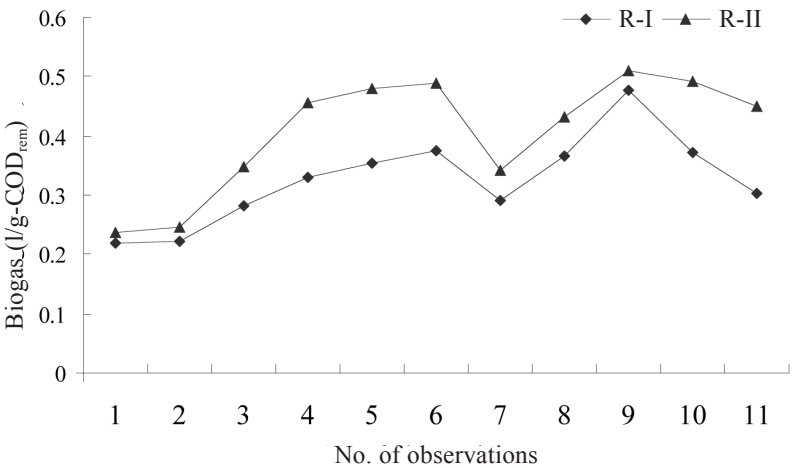

Figure 7. Amount of biogas production.

reactors R-I and R-II in the last weeks of the study period when the HRT was relatively low (below 12 h). The reason is that the lower HRT promotes the washout of sludge from the reactor and hence the amount of biogas production decreases by design.

Under optimum conditions, the average amount of biogas produced in the reactors R-I and R-II was observed to be 0.33 l/g $COD_{removed}$ and 0.41 l/g $COD_{removed}$, respectively. The average methane composition was slightly different for both the reactors R-I and R-II, i.e. 61% and 58%, respectively. The enhancement in the methane composition was mainly due to the adsorption of refractory material by activated carbon. It was also noticed that the reactor R-I displayed a reduced amount of biogas generation, which might be due to the low mixing of substrate and biomass owing to the presence of activated carbon within the system that made the sludge particles more dense. The overall gas production in both the reactors remained close to the theoretical value of 0.35 l/g $COD_{removed}$ which indicated the efficacy and consistency of the systems used in the study, as it has been previously reported that the presence of recalcitrant material within such kinds of waste caused reduction in biogas production (Arshad *et al.* 2009). Comparison between biogas productions under anaerobic condition for similar waste is also mentioned in Table 2.

Bio-kinetic Co-efficient

Puspendu (2008) reported that the Garu second-order model was the best fit model for a wide range of data sets in the UASB reactor. For this study, the kinetic constants of Y, k_d, k, K_s were determined by using the experimental data obtained during the course of study period to explore the performance appraisal of reactor R-I. The intercept line and the slope of line from the graph plotted between Lr and $1/\theta_c$ gave the values of k_d and Y, respectively. This plot is shown in Figure 8. Y was determined to be 0.26 g-VSS/g-TOC or 0.8 g-VSS/g-COD, while k_d was found to be 0.02 d^{-1}. The plot between 1/S and 1/Lr is shown in Figure 9. As shown, k and Ks were determined to be 0.7 g-TOC/g-VSS.d (about 1.9 g-COD/g-VSS.d) and 0.3 g-TOC/l, respectively.

The kinetic constants determined in this study were compared with similar work conducted by using only methanol, as the sole carbon source in the feeding solution, as shown in the Table 3. This table indicates that the presence of the use of actual paper mill effluent has negligible impact on the bio-kinetic constant of the reactors. The 'k' value appears to be

Table 2. Comparison of similar studies.

Substrate	Operational parameters	COD and AOX removal	Biogas production	Reference
Methanol	OLR = 21 g-COD/L-d	COD = 70%	0.30 l/g-$COD_{removed}$	Bhatti 1996
Synthetic waste (Chlorophenol)	OLR = 6.25 g-TOC/L.d HRT = 12–48 h	COD = 80%	0.13 l/g-$COD_{removed}$ CH_4 = 60%	Arshad & Hashim 2008
NSSC pulping effluent	OLR = 2.75 kg-COD/m^3d HRT = 38 h	COD = 35%	0.17 m^3/kg-COD_{rem}-d CH_4 = 61%	Arshad *et al*. 2009
Bleaching effluent (Paper mill)	OLR = 2.14 kg-COD/m^3-d HRT = 38 h	COD = 64% AOX = 49%	0.19 l/g-$COD_{removed}$ CH_4 = 58%–60%	Arshad & Hashim 2010
Paper mills effluent	OLR = 3.5 kg-COD/m^3-d HRT = 22 h	COD = 88% AOX = 72%	0.33 l/g-$COD_{removed}$ CH_4 = 60%–61%	This study

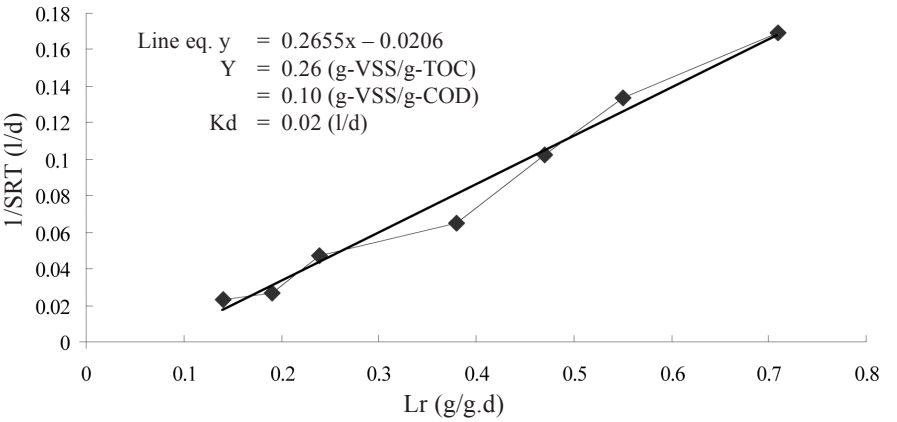

Figure 8. Relationship between specific subtrate removal rate and inverse of SRT.

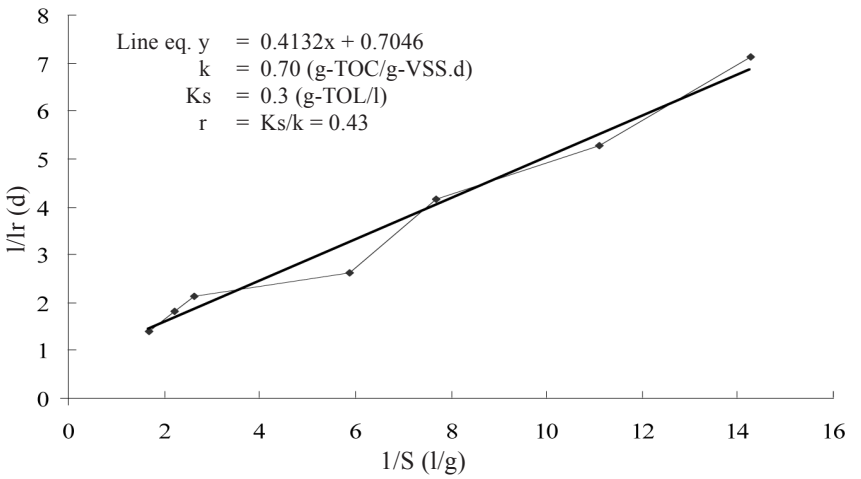

Figure 9. Determination of kinetic coefficiants of k and Ks.

reasonable, showing the good potential of the applicability of anaerobic treatment of paper mill effluent in the presence of activated carbon and methanol.

CONCLUSION AND RECOMMENDATIONS

The usage of methanol as an easily biodegradable substance in the presence of activated carbon with the digested sludge in a UASB reactor seems to be an extremely viable option for the treatment of paper mill effluent. The COD removal efficiency can be specifically enhanced from 64% to 88% and the AOX removal from 47% to 72%, at an OLR of 3.5 kg-COD/m^3-day. The optimum HRT for the UASB reactor design to treat such wastes under a mesophilic range of temperature and neutral pH was found to be 22 h.

The presence of activated carbon in the UASB reactor reduced the mixing of biomass and substrate, and consequently affected the formation of biogas. The gas production could be decreased from 0.41 l/g COD_{rem} to 0.33 l/g-COD_{rem} by using activated carbon within the

Table 3. Comparison of kinetic constants.

Type of waste	Y (g-VSS/g-TOC)	K_d (day^{-1})	Ks (g-TOC/L)	k (g-TOC/g-VSS.d)	Reference
Methanol	0.213	0.005	0.385	1.11	Bhatti *et al*. 1996
Paper mill effluent	0.26	0.02	0.30	0.70	This study

reactor. The average methane composition of biogas showed an increase from 58% to 61% by using activated carbon and methanol within the UASB reactor. Kinetic coefficients of k, K_s, Y and k_d for paper mills effluent treatment in UASB reactors were 0.7 g-TOC/g-VSS.d, 0.3 g-TOC/l, 0.26 g-VSS/g-TOC and 0.02 day^{-1}, respectively.

The feasibility of the treatment for paper mill effluent in a single-step UASB reactor in the presence of methanol, as an easily biodegradable substance, and activated carbon is a highly viable method but detailed study is essential to identify the exact behavior of the activated carbon and methanol during the process of digestion. Cost analysis for design of the UASB reactor needs to be evaluated on a mega scale if activated carbon is to be used, while the impact of variable pH and temperature also needs to be evaluated thoroughly.

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Physical and Mechanical Properties of Jute Mat Reinforced Epoxy Composites

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Cellulose jute fibre offers a number of benefits as reinforcement for synthetic polymers since it has a high specific strength and stiffness, low hardness, relatively low density and biodegradability. To reduce moisture uptake and hence to improve the mechanical properties of the composites, bleached jute mats were incorporated as reinforcing elements in the epoxy matrix. Composites at varying volume fractions and different orientations of jute mat were fabricated by hot compression machine under specific pressures and temperatures. Tensile, flexure, impact and water absorption tests of composites were conducted. Jute mat oriented at (0 ± 45–90)° composites showed reduced strength compared to (0–90)° fibre mat composites. Impact strength and water uptake of high volume fraction jute mat reinforced composites was higher compared to that of lower volume fraction composites. Fracture surfaces of jute mat composites were analyzed under SEM. Fracture surface of (0–90)° jute mat oriented composites showed twisted fibres, while (0 ± 45–90)° jute mat oriented composites had fibre pull-out without any twisting. Overall, composites containing 52% jute mat at orientations of (0–90)° showed better properties compared to other fabricated composites.

Key words: moisture reduction; SEM analysis; bleached jute mat; hot compression; pressure; temperature; tensile; flexure; water absorption; strength; fracture; fibre

Natural fibres exhibit many beneficial properties as reinforcements for composites. It is light in weight, cost effective and has high specific properties compared to synthetic fibres such as glass, carbon etc. Natural fibre reinforced composites are light in weight, possess better electrical resistance, good thermal and acoustic insulating properties and good resistance to fracture (Abu-Sharkh & Hamid 2004). Among all the natural fibre reinforcing materials, jute appears to be a promising material because it is relatively inexpensive and commercially available in the required form (Vilaseca *et al*. 2007; Planckett *et al*. 2003). It has higher strength and modulus compared to most of the plastics and is a good substitute for conventional fibres in many situations (Vilaseca *et al*. 2007; Planckett *et al*. 2003).

In this present work, jute fibre was used as the reinforcing material since it is produced in a large scale in the Indian sub-continent, especially in Bangladesh and has a minimal effect on the environment because of its biodegradable properties. Epoxy or polyepoxide, a thermosetting polymer formed from reaction of an epoxide 'resin' and with a suitable 'hardener' (hydrogen peroxide) was used as the matrix material. Thus, the aim of the study was to manufacture composites using raw bleached jute mat and epoxy, subsequently characterizing them using microstructural analysis and mechanical testing.

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EXPERIMENTAL PROCEDURE

Materials

The thermosetting co-polymer epoxy, used as matrix material, was supplied by the polyolefin Company Private Limited, Singapore. It had a specific gravity of 1.53. The jute, used as reinforcing fibre, was collected from Bangladesh Jute Research Institute (BJRI).

Fabrication of Composites and Preparation of Test Specimens

Four types of composites were manufactured by varying the number and orientation of jute mat ply. They were: (i) 4 ply in (0/90)° orientation; (ii) 4 ply in (0 ± 45–90)° orientation; (iii) 8 ply in 0/90° orientation and (iv) 8 ply in (0 ± 45–90)° orientation. The jute mat was first cut into 20 × 20 cm² sizes and dried in an oven at about 70°C. The required amount of resin and 0.1% hardener (H_2O_2) ware added and stirred properly. For the purpose of degassing the beaker, it was placed in desiccators which were connected to a vacuum pump. Degassing was carried out for 2 min – 3 min. The mould surface was cleaned very carefully and mould releasing agent (PVA, Wax) was sprayed thoroughly over the mould surface. Each ply was wet completely by the resin (epoxy). The ply was then passed through a roll mill for the removal of excess resin associated with the ply and to ensure it was rodden with resin. The ply was then placed in a female mould and then covered by a male mould. This arrangement was kept at 110°C temperature and 70 kN pressure under a hot pressure machine for about 1 h. The mould was then transferred to an oven and cured at 140°C–160°C for about 2 h and then cooled very slowly in the oven to complete curing. The mould specimen was carefully discharged from the mould.

Test specimens were fabricated individually to eliminate void conditions and to minimize edge and cutting effects during machining. Specimens were prepared to required dimensions according to ASTM standards [ASTM Standard D 638-01 (2002); ASTM Standard D 790-00 (2002); ASTM Standard D 6110-97 (2002); ASTM Standard D 570-99 (2002)].

Mechanical Testing

Tensile, flexure, impact and water absorption tests were subsequently conducted. Ten specimens of the type of composite were analysed for each test and the average values were recorded. Tensile tests were conducted according to ASTM-D 638-01 (2002) using a universal testing machine at a cross-head speed of 3 mm/min. The dimensions of the specimen were 120 mm × 10 mm × 4 mm. Three point static flexural tests were carried out according to ASTM D 790-00 (2002) using the same testing machine mentioned above. Dynamic Charpy impact tests were conducted using a universal impact testing machine following ASTM D 6110-97 (2002). Water absorption characteristics of the manufactured composites were measured according to ASTM D 570-99 (2002) using 2 h immersion.

Microstructural Analysis

Fractured surfaces of the tensile tested specimens were examined by using a scanning electron microscope (SEM) at a magnification of 200.

RESULTS AND DISCUSSION

Tensile Properties

Variation of tensile strength at different orientations and volume fractions (%) of jute mat are shown in Figure 1. Tensile strength of the composite was influenced by the tensile strength and modulus of the fibre. It was found that there was an increase in tensile strength with the incorporation of higher volume jute mat into epoxy matrix. The tensile properties were affected by the layering sequences. Tensile strength was higher for (0–90)° orientation of jute mat compared to (0 ± 45–90)° orientation. This was because the

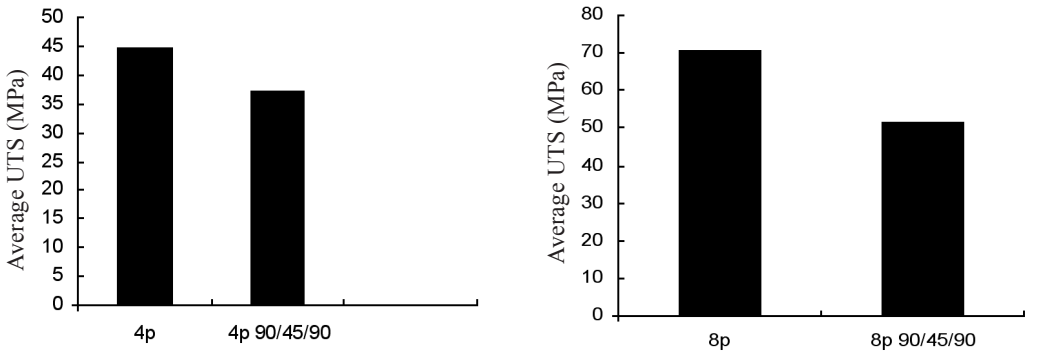

Figure 1. Variation of tensile strength at different orientation and volume fraction (%) of jute mat.

load applied was transferred through the fibre at 45° to the loading axis. Shearing action was predominant and hence the strength became somewhat lower.

Flexural Properties

Variation of flexural strength against fibre volume fraction (%) is shown in Figure 2. According to Figure 2, flexural strength initially decreased, then increased with increase in jute mat volume fraction (%).

Impact Strength Results

Figure 3 shows variation of Charpy impact strength against fibre volume fraction (%). The impact strength increased with fibre volume fraction (Lin *et al*. 2006; Joseph 2002; Jayaraman *et al*. 2003; Islam *et al*. 2009). The increase in impact strength of composites was due to the fact that the fibre was capable of absorbing energy because of the strong inter-facial bonding between the fibre and the matrix. Another factor for the impact failure of the composite was fibre pull out. With increase in fibre volume fraction, bigger force was required to pull-out the fibres. This consequently increased the impact strength.

Water Absorption Characteristics

The water absorption characteristics of the manufactured composites against time at

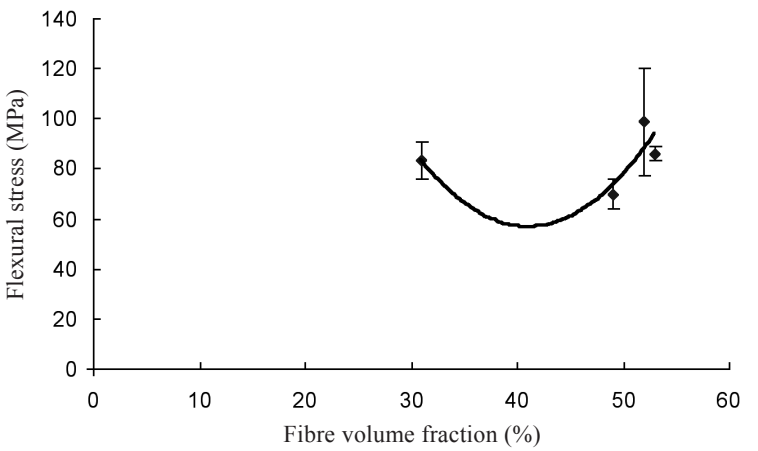

Figure 2. Variation of flexural strength against jute mat volume fraction (%).

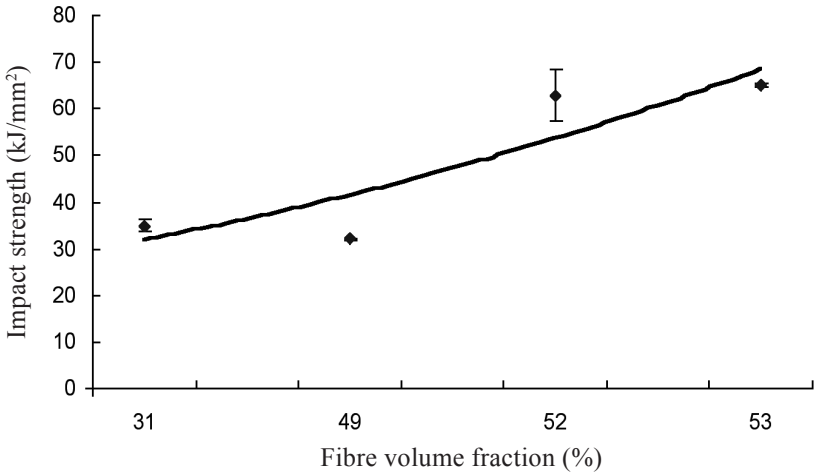

Figure 3. Variation of impact strength against jute mat volume fraction (%).

2 types of jute mat ply and orientation, that is for 4 ply in (0 ± 45–90)° and 8 ply in (0 ± 45–90)° orientation are shown in Figures 4 and 5 respectively. The water absorption (%) increased with the increase in fibre loading (Yang *et al*. 2006; Matuana *et al*. 2001). The hydroxyl group in jute mat is mainly responsible for water absorption. With increase in jute fibre volume fraction, the number of hydroxyl groups in the composite increased, which subsequently increased water absorption. Figures 4 and 5 also show that with an increase in time, water absorption increased.

SEM Morphology

Figure 6 shows the SEM fracture surface of 4 ply jute mat composite in (0–90)° fibre orientation, while *Figure 7* shows the SEM fracture surface of 4 ply jute mat composite in (0 ± 45–90)° fibre orientation. The fracture surface of (0–90)° jute mat oriented composites showed twisted fibre (*Figure 6*), which meant the fibres were twisted during the tensile test. On the other hand, (0 ± 45–90)° jute mat oriented composites had fibres pulled out without any twisting (*Figure 7*) that meant that the fibres were pulled out from the composites during tensile loading.

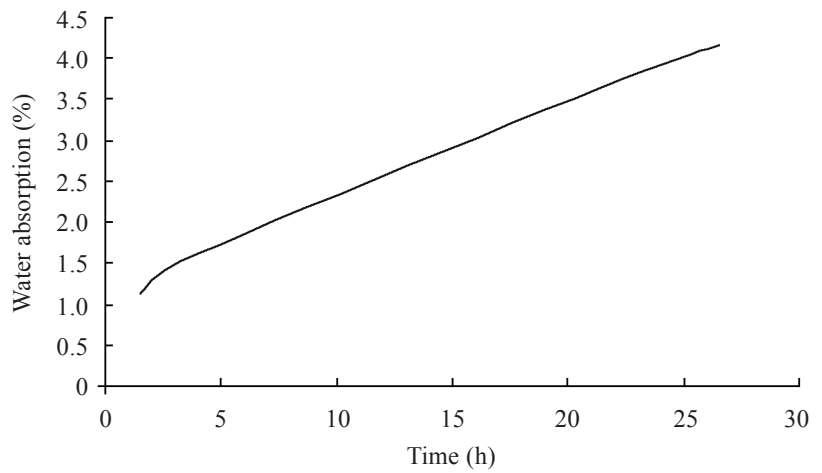

Figure 4. Variation of water absorption (%) against time for 4 ply in (0 ± 45–90)° orientation.

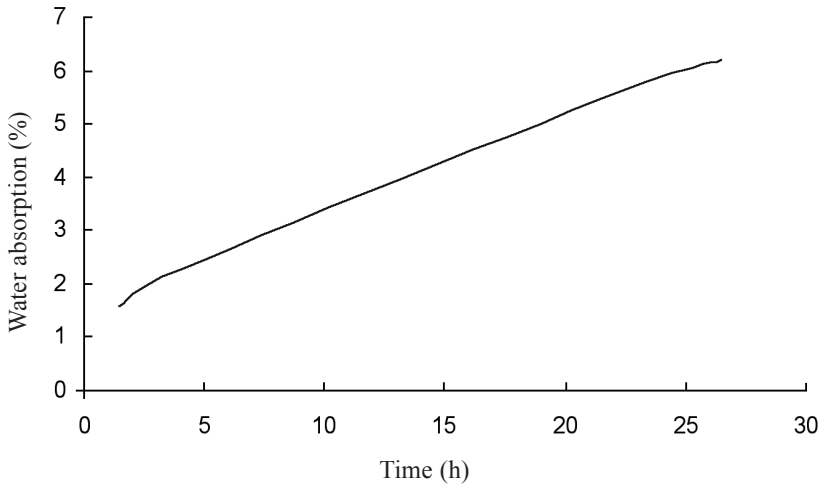

Figure 5. Variation of water absorption (%) against time for 8 ply in (0 ± 45–90)° orientation.

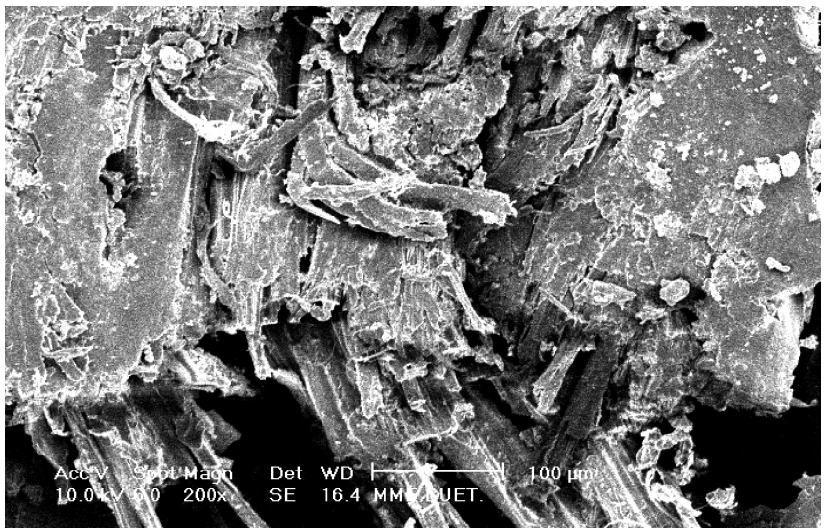

Figure 6. Fractured surface of 4 ply composite in (0–90)° fibre orientation.

CONCLUSION

Bleached jute mat reinforced-epoxy composites of varying volume fractions (%) and at different orientations of jute mat were fabricated using the hot compression machine under specific pressure and temperature. Tensile, flexure, impact, water absorption of composites and scanning electron micrography of fracture surface of composites were conducted. Jute mat oriented at (0–90)° composites had a higher tensile strength compared to (0 ± 45–90)° fibre mat composites. Impact strength and water uptake increased with increase in jute mat fraction. Fracture surfaces of jute mat composites were analyzed under SEM. Fracture surface of (0–90)° jute mat oriented composites had twisted fibres, while (0 ± 45–90)° jute mat oriented composites had fibre pull-out without any twisting. Considering the experimental results, composites containing 52% jute mat at orientations of (0–90)° had better properties compared to other fabricated composites. Thus

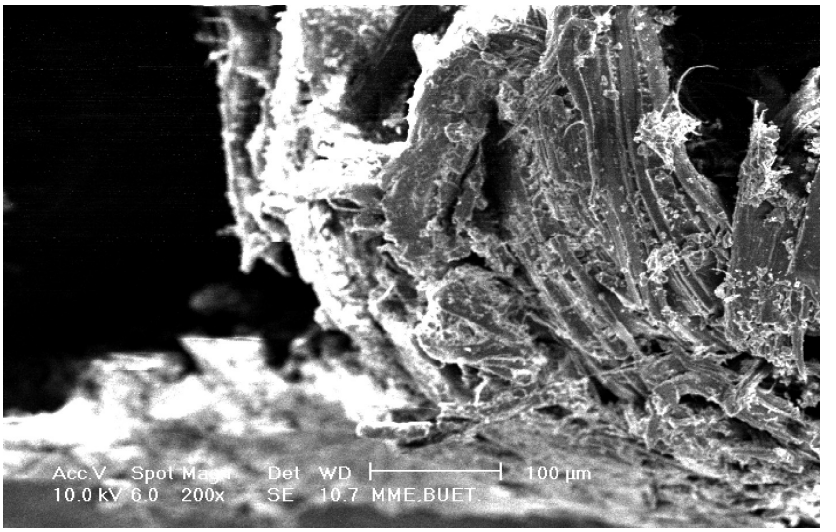

Figure 7. Fractured surface of 4 ply composite in (0 ± 45–90)° fibre orientation.

it would be better to manufacture composites with 52 volume fraction (%) jute mat at (0–90)° orientations to obtain optimum utilization during service.

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Nutrient Composition of *Artocarpus champeden* and Its Hybrid (*Nanchem*) in Negara Brunei Darussalam

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The flesh and seeds of ripened and unripened *Artocarpus champeden* and its ripened hybrid (*Nanchem*) were analyzed for their moisture, ash, crude fibre, crude protein, crude fat, total carbohydrate, energy and mineral content. Generally, unripened *A. champeden* which is always treated and cooked as a vegetable contains higher amounts of moisture, ash, crude fibre and crude protein for its flesh than ripened *A. champeden* and *Nanchem*. Ripened *A. champeden* and *Nanchem* have higher total carbohydrates and energy content than the unripe fruit. Similarly, the unripened *A. champeden* seed has more nutritional components in terms of moisture, ash, crude fibre, crude protein, crude fat, total carbohydrate and energy compared to the ripened *A. champeden* and *Nanchem* seeds. Potassium and magnesium are the prevalent minerals in this fruit species. Nanchem has the characteristics of both jackfruit (*A. heterophyllus*) and *A. champeden*

Key words: *Artocarpus champeden*; *Tibadak*; *Nanchem*; ripened; unripened; moisture; crude fibre; crude protein; crude fat; total carbohydrate; energy; mineral content

Artocarpus champeden (Thunb.) *Merr* (*syn Artocarpus integer Merr.*) belongs to the Moraceae family. The name of the genus *Artocarpus* is derived from the Greek words *artos* which means bread and *carpos* which means fruit (Bailey 1942). *A. champeden* is in the same family as jackfruit (*A. heterophyllus* Lam.), breadfruit (*A. altilis*) and tarap (*A. odoratissimus*) (Janick & Paull 2008; Subhadrabandhu 2001). The fruit of this genus is generally large (Figure 1).

A. champeden is traditionally grown in tropical and sub-tropical regions, particularly in Southeast Asia where it is widely distributed in southern Thailand, Peninsular Malaysia, Myanmar, Vietnam and Indonesia (Subhadrabandhu 2001). Consequently, it has various vernacular names depending on the country and language; such that in Thailand it is called '*Champada*' and in Myanmar it is known as '*Sonekadat*' (Janick & Paull 2008; Jarrett 1960). In Brunei Darussalam, the fruit is locally known as either '*Tibadak*' or '*Cempedak*'.

The *A. champeden* fruit (Figure 1) can be consumed either ripe or unripe (mature or immature). Both the flesh and seed (Figure 2) of *A. champeden* are edible; the skin and core are inedible and are discarded as waste. The flesh is normally eaten fresh, deep fried into fritters, processed into a refreshing juice, dried into chips or creamed to make jams and cakes. Unripe *A. champeden* is always treated as a vegetable and cooked in coconut milk, eaten along with vegetables, or in soup: a common delicacy in Malaysia and south India (Janick & Paull 2008; Subhadrabandhu 2001). The seed is either roasted or boiled in salty water; it is a popular delicacy amongst the Malayan jungle tribes (Janick & Paull 2008; Subhadrabandhu 2001). Moreover, the seed can

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Figure 1. Artocarpus champeden *fruit (Length:23 cm).*

Figure 2. Artocarpus champeden *flesh and seed with Brunei 50 cent coin (diameter: 28 mm) for comparison.*

be dried and ground to make flour for baking. *A. champeden*'s seed flour provides a good source of dietary fibre and resistant starch (Zabidi & Aziz 2009).

Research has shown that the skin of the *A. champeden* fruit can be used for the removal of methylene blue (Prahas *et al*. 2008) and cadmium(II) ions from aqueous solution (Inbaraj & Sulochana 2004). On the other hand, the stem bark, aerial plant and roots have been reported to possess anti-malarial properties (Boonlaksiri *et al*. 2000) and cytotoxicity (Hakim *et al*. 2006; Hakim *et al*. 2005). *A. champeden* seeds contain lectins such as IgA1-reactive and $_{D}$-galactose-binding lectin. These lectins have been found to be useful in biomedical research for the detection of tumors and the identification of glycoprotein (Lim *et al*. 1997).

A. champeden is commonly grown from the seed; with this method the tree would only bear fruit after five years. Grafting is another method that is widely used in agriculture where plant tissue such as the stem or bark are fused with another plant. With the success of this method, many variations of *A. champeden* can be found in the market nowadays. One such popular variation commonly found in Brunei Darussalam is its hybrid with *A. heterophyllus* (jackfruit). The hybrid is locally called '*Tibadak-Nangka*' or '*Nanchem*' since the local name for jackfruit is *Nangka* and *A. champeden* is known as *Chempedak*. The *Nanchem* fruit is larger and sweeter; the flesh is larger (Figure 3) and it has an attractive intense orange colour. Both *A. champeden* and its hybrid are popular edible fruits in Brunei Darussalam as well as in South East Asia due to their soft and firmly textured flesh.

Figure 3. Nanchem *flesh, seed and a 50 cent coin (diameter: 28 mm) for comparison.*

Various studies have been conducted on the *A. champeden* relative, *A. heterophyllus* (jackfruit). However, only a limited number of studies have been done on *A. champeden* flesh and seed. Janick and Paull (2008) have reported on the nutrient composition of *A. champeden* flesh, but the origin of the sample was not stated. Subhadrabandhu (2001) studied *A. champeden* flesh in Thailand, while Zabidi and Aziz (2009) have studied *A. champeden* seed in Malaysia.

The aim of this study was therefore to carry out a proximate analysis of the *A. champeden* (ripe and unripe) found in Brunei Darussalam together with its hybrid.

MATERIALS AND METHODS

All the reagents and solvents were of analytical grade and obtained from Sigma-Aldrich or Merck.

Instrumentation

The following instruments were used: A *VIRTIS Specimen* freeze dryer for freeze drying, a *Thermolyne 1400* muffle furnace for ash, a *FOSS Fibertec™ 2010* for crude fibre, a *Gerhardt* automated distillation system and Kjeldhal digestion machine for the determination of crude protein, a *Shimadzu* UV-1601pc UV-VIS spectrophotometer for total carbohydrate, a *Gallenkamp* auto bomb calorimeter for the energy and a Shimadzu AA-*6701F* atomic absorption flame emission spectrophotometer for the mineral analysis. All analyses were a modification of the AOAC official method (Cunniff 1998) and carried out at least in duplicate.

Sample Collection

All the fruit samples of *A. champeden* (ripe and unripe) and its hybrid (*Nanchem)* were purchased from the local wet markets in Bandar Seri Begawan, the capital of Brunei Darussalam, during July and August 2009. Nine fruits were bought for the ripe *A. champeden*, six fruits for the unripe *A. champeden* and nine fruits for the *Nanchem*.

Sample Preparation

The fruits were weighed and immediately cut open (Figure 4). The fruits were then separated into flesh, seed, skin and core. The fruits were categorized according to their flesh colour and skin texture (spikiness). The categorized parts were then weighed and kept in freezer at –20°C in pre-cleaned polyethylene bags prior to analysis. The samples were extracted using the AOAC Official method 920.149 (Cunniff 1998).

Figure 4. Cut open fruit of ripe A. champeden.

Moisture Content

Oven drying. Samples (10 g) were cut into smaller pieces and spread evenly across a pre-weighed drying dish. Flesh and seed samples were dried to a constant mass at 50°C while the skin and core were dried at 80°C. The lower temperature was used for the flesh and seed samples in order to minimize the release of volatile components. After heating, the sample was cooled in a desiccator. Upon obtaining a constant weight, the dried sample was ground into a fine powder using a Phillips blender and stored in pre-cleaned polyethylene bags in a desiccator (Kirk & Sawyer 1991; Ranganna 1986).

Freeze drying. Weighed *A. champeden* and *Nanchem* flesh samples were freeze-dried at –74°C at a vacuum of 150 – 200 millibar to constant mass; the dried samples were then stored in a desiccator.

Ash

The fresh samples (10 g) in silica crucibles were heated in a muffle furnace at 600°C for 5 h (Kirk & Sawyer 1991; Ranganna 1986). The ash content on a fresh weight basis was thus converted to a dry weight basis.

Energy

The energy content of the oven dried samples (flesh and seed) was determined using a bomb calorimeter; benzoic acid was used as the standard.

Crude Fibre

Determination was carried out using 2 g of dried sample. Sulphuric acid (1.25%, 200 ml) was added and the sample was boiled for exactly 30 min. The sulphuric acid was drained off by vacuum filtration and the sample was washed with near boiling water until traces of acid were undetected by pH paper. Near boiling point sodium hydroxide (1.25%, 200 ml) was added into the sample followed by 2 drops of octanol and boiled for exactly 30 min. The sodium hydroxide was drained off by vacuum filtration. The residue was washed with near boiling water (50 ml) followed by 1.25% sulphuric acid (30 ml) and lastly washing was repeated with near boiling water (60 ml). The digested sample was oven-dried at 130°C overnight. The dried

sample was ashed in a muffle furnace for 4 h at 550°C. (Madamba 2000; Ranganna 1986).

Crude Protein

Nitrogen was determined using the modified Kjeldahl method. The dried sample (1 g), a Kjeldhal tablet with a selenium catalyst and concentrated sulphuric acid (10 ml) were digested for 2 h using a Gerhardt Kjeldhal digestion machine. The distillation and titration were carried out using Gerhardt distillation system and the percentages of nitrogen were converted to protein by multiplying by 6.25 for the flesh, skin and core and 5.3 for the seed (Kirk & Sawyer 1991; Nielsen 2003b).

Crude Fat

Crude fat was determined by Soxhlet extraction of the dried ground flesh (10 g) and seed (10 g) samples with *n*-hexane (150 ml) for 6 h. The *n*-hexane was removed by rotary evaporation and the yellow oil was weighed (Nielsen 2003a).

Total Carbohydrate

This analysis was carried using the phenol-sulphuric acid method (AOAC Method 44.1.30), as stated in the Food Analysis Laboratory Manual (BeMiller 2003; Nielsen 2003c) with slight modification. The standard was prepared by dissolving glucose (0.01 g) in doubly distilled water (100 ml).

Fresh samples (5g to 20 g) were homogenized using a *Philips* blender in doubly distilled water (100 ml). Known volumes of the homogenized sample were further diluted. For a ripened *A. champeden* and *Nanchem* flesh (0.15 ml or 0.25 ml), unripened *A. champeden* (1 ml or 3 ml) and the seed (1 ml), aliquots were transferred to a 100 ml volumetric flask and topped up using doubly distilled water. The diluted sample (1 ml) was transferred into a test tube containing doubly distilled water (1 ml). An 80% phenol solution (50 µl) was added into the standards and samples followed by concentrated sulphuric acid (5 ml), whereupon the colourless solution immediately became yellowish orange. The absorbance of the standards and samples were immediately recorded at 490 nm.

Mineral Analysis

Concentrated hydrochloric acid (2.5 ml) and concentrated nitric acid (2.5 ml) were added to the ash. The dissolved ash was transferred into a 25 ml volumetric flask and topped up to the mark with ultra pure water. The solutions were gravity filtered using Whatman 41 ashless filter paper into pre-cleaned plastic bottles.

RESULTS AND DISCUSSION

The proximate analysis of the ripened and unripened *A. champeden* and ripened *Nanchem* was determined for the edible portions of the fruit, viz. the flesh and seed. The analyses performed were mass composition, moisture, ash, crude fiber, crude protein, crude fat, total carbohydrate, energy and the mineral content. The results for ripened and unripened *A. champeden* and *Nanchem* flesh and seed were on a dry weight basis except for total carbohydrate which was done on a fresh weight basis.

Physical Description

A. champeden fruit weights from 1 kg to 3.5 kg and the average length of the fruit is 23 cm which is smaller with *A. heterophyllus* (jackfruit). Both *A. champeden* and *A. heterophyllus* (jackfruit) have a green, yellow or brown skin that is divided into small hexagons and the texture of the skin is either smooth or spiky (Figure 1). Like *A. heterophyllus* (jackfruit), the colour of the flesh is golden yellow to orange while the seed is brown in colour and is surrounded by the yellow flesh (Figure 2). The texture of the *A. champeden* flesh is soft, while for *A. heterophyllus* (jackfruit) the flesh is crunchy. Both *A. champeden* flesh

and seed are edible, but its skin and core are normally discarded.

Nanchem fruit weighs from 1 to 5 kg and the fruit average length is about 30 cm. The shape of the fruit is elongated like *A. champeden*. It has similar skin texture and flesh color with *A. champeden* and *A. heterophyllus*. However, *Nanchem*'s skin is spikier (Figure 5) than *A. champeden* and divided into small pyramidal hexagons. When *Nanchem* is cut opened (Figure 4), the flesh is attached to a light brown colored core which is hairy and is about 10 cm in length. *Nanchem* flesh is thicker and is bright orange or yellow in color (Figure 3). The seed is surrounded by its flesh. The seed on the other hand is smaller than *A. champeden* and *A. heterophyllus*.

Mass Composition

The average amount of flesh in ripe *A. champeden* was 26.5% (9 fruits) while that for unripe *A. champeden* (6 fruits) and *Nanchem* (9 fruits) is 24.4% each. *Nanchem* flesh was more fibrous and juicier than the ripe *A. champeden*; this might account for its popularity in Brunei Darussalam.

Table 1 shows the mass composition of the samples. The seed content in ripe and unripe *A. champeden* and *Nanchem* are 31.4%, 16.9% and 6.8% respectively. The ripe *A. champeden* has the most seeds with larger seeds compared to *Nanchem* (Figure 2 and Figure 3).

The skin and core are inedible and are always treated as waste in Brunei Darussalam. The skin is the heaviest part of the fruit, while the core is the lightest part. *Nanchem* has more skin than *A. champeden.* However, unripe *A. champeden* has more skin than its ripe specimens. Since the fruit is usually traded based on its weight, the *Nanchem* has the least value for money as the inedible portion (skin and core) amounts to almost 69% of the whole fruit compared to only 42% for the ripe *A. champeden*.

As the *Nanchem* fruit consists of about 70% skin, it would be profitable if the skin could be utilized. For example, it has been shown that *A. heterophyllis* (jackfruit) skin removes methylene blue (Prahas *et. al.* 2008) and cadmium(II) ions (Inbaraj & Sulochana 2004) from aqueous solution. Therefore, *Nanchem* skin could be used as a bio-sorbent to absorb the impurities in water rather than being disposed as waste. On the other hand, the seed that is normally eaten boiled could be used in baking replacing wheat flour like *A. heterophyllus* (jackfruit) (Zabidi & Aziz 2009).

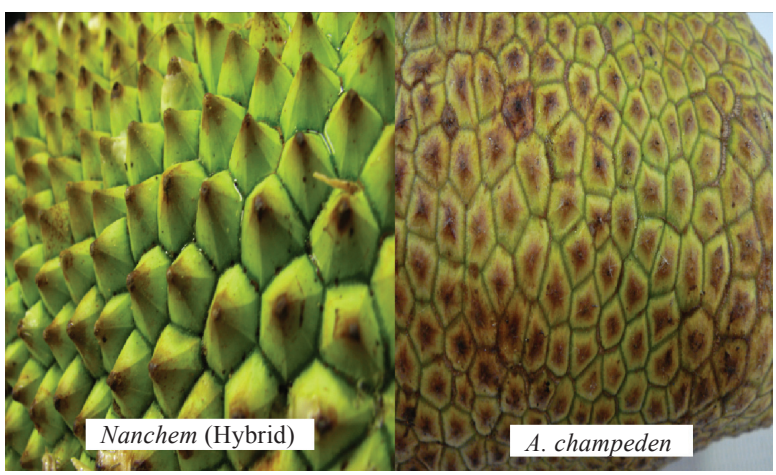

Figure 5. Nanchem *(hybrid) (left) and* A. champeden *(right) skin texture.*

Table 1. Mean mass composition (%) of *A. champeden* and *Nanchem* fruit.

Type		n[a]	Flesh	Seed	Skin	Core
Champeden	Ripe	9	26.5	31.4	36.8	5.4
	Unripe	6	24.4	16.9	52.4	6.4
Nanchem	Hybrid	9	24.4	6.8	63.5	5.3

[a] n represents the number of samples.

Moisture Content

The moisture content of the fruit was determined by both oven-drying and freeze-drying. The moisture content in ripe and unripe *A. champeden* flesh was in the range of 62.3% – 73.4% and 82.7% – 88.1%, respectively. For its hybrid flesh, the moisture content was within the range of 59.7% – 69.5%. The range 58% – 87% has been reported by Janick & Paull (2008) and Subhadrabandhu (2001) in Thailand. The moisture in *Nanchem* flesh was lower than *A. heterophyllus* (jackfruit) (72 – 94%) and was similar to *A. champeden*. Unripe *A. champeden* had the highest moisture content which was similar to the moisture content in vegetables (80% – 90%) (Kirk & Sawyer 1991).

The moisture content in the seeds of ripe and unripe *A. champeden* and *Nanchem* was in the range 52.9 – 91.1%. The range 46% – 78% has been reported by Subhadrabandhu (2001) and Zabidi & Aziz (2009). Unripe *A. champeden* (83.6% – 91.1%) contained the highest percentage of moisture in its seed. Ripe *A. champeden*'s seed (52.9% – 72.8%) contained more moisture than the *Nanchem* seed (49.7% – 54.0%), but lower than the highmoisture content in *A. heterphyllus* (jackfruit) (64%). Water in fruit plays a part in controlling the microbial activity and high moisture content will reduce the shelf life of a particular fruit (Chowdhury *et al*. 1997). The order for moisture content in ripe *A. champeden* and *Nanchem* was: flesh > seed and no trends was evident for unripe *A. champeden*.

Figure 6 shows that both drying methods yield similar moisture content for the samples (ripe *A. champeden* and *Nanchem*). In spite of this, the appearance of the dried samples were dissimilar. For oven dried, the sample turned from a yellow to a dark-brown colour

Table 2. Proximate analyses of *A. champeden* and *Nanchem* flesh and seeds[a].

Analysis (%)	Flesh			Seed		
	A. champeden		*Nanchem*	*A. champeden*		*Nanchem*
	Ripe	Unripe		Ripe	Unripe	
Moisture	62.3 – 73.4	82.7 – 88.1	59.7 – 69.5	52.9 – 72.8	83.6 – 91.1	49.7 – 54.0
Ash	2.5 – 3.9	4.6 – 5.0	2.2 – 2.7	3.2 – 5.1	4.0 – 5.0	2.8 – 3.2
Crude Fibre	4.6 – 7.6	12.9 – 23.9	2.5 – 3.3	3.9 – 7.1	8.0 – 8.3	4.7 – 8.5
Crude Protein	4.9 – 5.8	7.3 – 15.9	4.1 – 7.5	9.9 – 11.2	12.1 – 17.9	8.9 – 10.9
Crude Fat	2.4 – 3.5	3.9 – 6.4	0.8 – 4.1	0.8 – 2.4	1.5 – 2.3	0.9 – 1.7
Total Carbohydrate (g/100 g)	16.2 – 28.3	2.4 – 5.1	7.5 – 30.0	2.8 – 3.5	1.8 – 2.5	3.2 – 3.4
Energy (kcal/100g)	430 – 437	456 – 463	456 – 477	431 – 447	467 – 539	465 – 480

[a] All analyses were performed in dry samples, except for total carbohydrate which was carried out on a fresh weight basis.

after drying while for freeze dried, the sample retained its original colour and odour. The contributing factors for the change in colour on heating are the Maillard reaction and enzymatic reactions (Chong *et al*. 2009). The texture of the sample turned hard and springy when oven-dried.

According to Chong *et al.* (2009), the quality of the dried fruit is higher if it is soft. Hence, the freeze-drying method is believed to produce a higher quality of dried sample due to the soft dried fruit obtained after drying, and this technique protects the primary structure of the sample by solidifying the water. On the other hand, the springiness in oven-drying sample is due to the gelling agents in the fruits such as pectin. A high temperature could induce the pectin substances in fruit to restructure and therefore cause the oven-dried sample to be hard and springy. In terms of time however, oven-drying was preferable.

Ash

The ash in the flesh of ripe *A.champeden* and *Nanchem* was 2.5% – 3.9% and 2.2% – 2.7%, respectively. These results were similar to the reported values of *A. champeden*: 1.2% (Janick & Paull 2008) and 2% – 4% (Subhadrabandhu 2001) (Table 3). Unripe *A. champeden* had more ash (4.6% – 5.0%) compared to the ripe fruit and *Nanchem*.

For the seeds, both ripe *A. champeden* and *Nanchem* had similar ash content with the reported values (2.6% – 4.0%) (Subhadrabandhu 2001; Zabidi & Aziz 2009) (Table 4). Unripe *A. champeden* seed (4.0% – 5.0%) had more ash than the ripe *A. champeden* (3.2% – 5.1%) and *Nanchem* (2.8% – 3.2%). For ripe *A. champeden* and *Nanchem*, the amount of ash in the seed was higher than that in the flesh. However, unripe *A. champeden* had a similar amount of ash for both flesh and seed.

Crude Fibre

Unripe *A. champeden* flesh and seeds had more crude fibre than the ripe fruit and the hybrid species (Table 2). Ripe and unripe *A. champeden* flesh contained more crude fibre than the seed while *Nanchem* seed has more crude fibre than its flesh. Unripe *A. champeden* flesh (12.9% – 23.9%) had the highest amount of crude fibre followed by ripe *A. champeden* (4.6% – 7.6%) and *Nanchem* (2.5% – 3.3%). Crude fibre values of 3.4% and 5% – 6% were reported by Janick & Paull (2008) and Subhadrabandhu (2001) (Table 3), respectively for *A. champeden* flesh. Generally, the order for ripe and unripe *A. champeden* was: flesh > seed, whereas the order for *Nanchem* was: seed > flesh.

For seeds, the crude fibre value reported by Zabidi & Aziz (2009) (2.44%) (Table 4) is lower

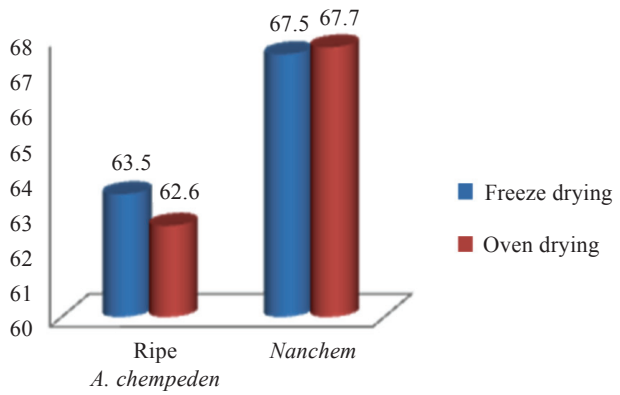

Figure 6. Moisture content (%) of the oven dried and freeze dried flesh.

for *A. champeden* flesh. Generally, the order for ripe and unripe *A. champeden* was: flesh > seed, whereas the order for *Nanchem* was: seed > flesh.

For seeds, the crude fibre value reported by Zabidi & Aziz (2009) (2.44%) (Table 4) is lower than the values obtained in this study. However, the value reported by Subhadrabandhu (2001) is 4% – 6% which was similar to the ripe *A. champeden* and *Nanchem*. On the other hand, crude fibre value for *A. heterophyllus* reported by Haq (2006) was 1.3% which was lower than ripe *A. champeden* and *Nanchem* seeds.

Crude Protein

Unripe *A. champeden* flesh was the highest source of crude protein (7.3% – 15.9%) compared with *Nanchem* (4.1% – 7.5%) and ripe *A. champeden* (5.0% – 5.8%). In terms of the ripe species, *Nanchem* contained slightly more crude protein than *A. champeden*. Janick & Paull (2008) and Subhadrabandhu (2001) reported values in the range of 2.5% – 7.0% (Table 3). *Nanchem* flesh had a similar amount of crude protein as the reported value for ripe *A. champeden* (2.5% and 3.5% – 7.0%) (Janick & Paull 2008; Subhadrabandhu 2001), but higher than *A. heterophyllus* (jackfruit) (1.6%) (Janick & Paull 2008). In terms of flesh, the order was: unripe *A. champeden* > *Nanchem* > ripe *A. champeden*.

Generally, the seed contains more crude protein than its flesh; this is quite reasonable as most seed samples are a rich source of protein (Zabidi & Aziz 2009). Similar to its flesh, unripe *A. champeden* seed (12.1% – 17.9%) contained more crude protein than ripe *A. champeden* (9.9% – 11.2%) and *Nanchem* (8.9% – 10.9%). This was a reverse for *Nanchem* as the seed had a lower percentage of crude protein than the ripe *A. champeden*. Therefore, the order of crude protein for the seed was: unripe *A. champeden* > ripe *A. champeden* > *Nanchem*.

Crude protein of *A. champeden* seeds reported by Subhadrabandhu (2001) and Zabidi & Aziz (2009) are 10% – 13% and 12.88% (Table 4), respectively. These values were similar to the amount of crude protein in ripe *A. champeden* and *Nanchem*. However, unripe *A. champeden* seeds contained more crude protein than ripe *A. champeden*, *Nanchem* and the values reported by Subhadrabandhu (2001), and Zabidi and Aziz (2009). Since *Nanchem* is the hybrid of *A. champeden* and *A. heterophyllus* (jackfruit), a comparison between *A. champeden* and *Nanchem* was made. *Nanchem* seeds contained more crude protein than *A. heterophyllus* (jackfruit) (6.6%) and its value was more similar to that of ripe *A. champeden*.

Crude Fat

For the flesh, the percentage of crude fat was slightly higher than the reported values (0.4% – 2.0%) (Janick & Paull 2008; Subhadrabandhu 2001) (Table 3) for all species. For example, the percentage of crude fat for ripe and unripe *A. champeden* and *Nanchem* was in the range of 2.4% – 3.5%, 3.9% – 6.4% and 0.8% – 4.1%, respectively. Unripe *A. champeden* flesh contained more crude fat than the ripe *A. champeden* which contained more crude fat than its hybrid (*Nanchem*). Generally, the order of crude fat for flesh was: unripe *A. champeden* > ripe *A. champeden* > *Nanchem*.

The crude fat for ripe and unripe *A. champeden* and *Nanchem* seeds was 0.8% – 2.4%, 1.5% – 2.3% and 0.9% – 1.7% respectively. The crude fat reported by Subhadrabandhu (2001) on *A. champeden* seed is 0.5% – 1.5% (Table 4) while the crude fat in *A. heterophyllus* (jackfruit) seed was only 0.4%. Ripe *A. champeden* contained somewhat more crude fat than the unripe and hybrid species. The seeds had less crude fat than the flesh; the order of crude fat for seed samples was: ripe *A. champeden* > unripe *A. champeden* > *Nanchem*.

Table 3. Proximate analysis of *A. champeden* (ripe and unripe) and *Nanchem* flesh: Comparison of the present study with reported values on a dry weight basis (except ash and total carbohydrate).

Analysis (%)	Previous studies			Present study		
	A. champeden		*A. heterophyllus*	*A. champeden*		*Nanchem*
	Janick & Paull[a]	Subhadrabandhu[c]	Janick & Paull[a]	Ripe	Unripe	
Edible portion	22	25–50	28	24.1–28.7	22.8–26.0	9.8–35.9
Moisture	87	58–85	83	62.3–73.4	82.7–88.1	59.7–69.5
Ash	1.2	2–4	2.2	2.5–3.9	4.6–5.0	2.2–2.7
Crude fibre	3.4	5–6	5.6	4.6–7.6	12.9–23.9	2.5–3.3
Crude protein	2.5	3.5–7.0	1.6	4.9–5.8	7.3–15.9	4.1–7.5
Crude fat	0.4	0.5–2.0	0.2	2.4–3.5	3.9–6.4	0.8–4.1
Total carbohydrate (g/100 g)[b]	25.8	84–87	9.4 – 25.4	16.2–28.3	2.4–5.1	7.5–30.0
Energy (kcal/100 g)	490	–	301	430–437	456–463	456–477
Mineral (mg/100 g)						
Calcium	40	–	37	3.4	5.1	2.6
Iron	1.1	–	1.7	0.5	0.5	0.4
Potassium	246	–	292	434	288	302
Sodium	25	–	48	1.1	0.8	1.6

[a] Reported value did not specify fresh or dry weigh basis
[b] Analysis carried out on fresh sample
[c] Values reported on dry weight basis

than the values obtained in this study. However, the value reported by Subhadrabandhu (2001) is 4% – 6% which was similar to the ripe *A. champeden* and *Nanchem*. On the other hand, crude fibre value for *A. heterophyllus* reported by Haq (2006) was 1.3% which was lower than ripe *A. champeden* and *Nanchem* seeds.

Crude Protein

Unripe *A. champeden* flesh was the highest source of crude protein (7.3% – 15.9%) compared with *Nanchem* (4.1% – 7.5%) and ripe *A. champeden* (5.0% – 5.8%). In terms of the ripe species, *Nanchem* contained slightly more crude protein than *A. champeden*. Janick & Paull (2008) and Subhadrabandhu (2001) reported values in the range of 2.5% – 7.0% (Table 3). *Nanchem* flesh had a similar amount of crude protein as the reported value for ripe *A. champeden* (2.5% and 3.5% – 7.0%) (Janick & Paull 2008; Subhadrabandhu 2001), but higher than *A. heterophyllus* (jackfruit) (1.6%) (Janick & Paull 2008). In terms of flesh, the order was: unripe *A. champeden* > *Nanchem* > ripe *A. champeden*.

Generally, the seed contains more crude protein than its flesh; this is quite reasonable as most seed samples are a rich source of protein (Zabidi & Aziz 2009). Similar to its flesh, unripe *A. champeden* seed (12.1% – 17.9%) contained more crude protein than ripe *A. champeden* (9.9% – 11.2%) and *Nanchem* (8.9% – 10.9%). This was a reverse for *Nanchem* as the seed had a lower percentage of crude protein than the ripe *A. champeden*. Therefore, the order of crude protein for the seed was: unripe *A. champeden* > ripe *A. champeden* > *Nanchem*.

Crude protein of *A. champeden* seeds reported by Subhadrabandhu (2001) and Zabidi & Aziz (2009) are 10% – 13% and 12.88% (Table 4), respectively. These values were similar to the amount of crude protein in ripe *A. champeden* and *Nanchem*. However, unripe *A. champeden* seeds contained more crude protein than ripe *A. champeden*, *Nanchem* and the values reported by Subhadrabandhu (2001), and Zabidi and Aziz (2009). Since *Nanchem* is the hybrid of *A. champeden* and *A. heterophyllus* (jackfruit), a comparison between *A. champeden* and *Nanchem* was made. *Nanchem* seeds contained more crude protein than *A. heterophyllus* (jackfruit) (6.6%) and its value was more similar to that of ripe *A. champeden*.

Crude Fat

For the flesh, the percentage of crude fat was slightly higher than the reported values (0.4% – 2.0%) (Janick & Paull 2008; Subhadrabandhu 2001) (Table 3) for all species. For example, the percentage of crude fat for ripe and unripe *A. champeden* and *Nanchem* was in the range of 2.4% – 3.5%, 3.9% – 6.4% and 0.8% – 4.1%, respectively. Unripe *A. champeden* flesh contained more crude fat than the ripe *A. champeden* which contained more crude fat than its hybrid (*Nanchem*). Generally, the order of crude fat for flesh was: unripe *A. champeden* > ripe *A. champeden* > *Nanchem*.

The crude fat for ripe and unripe *A. champeden* and *Nanchem* seeds was 0.8% – 2.4%, 1.5% – 2.3% and 0.9% – 1.7% respectively. The crude fat reported by Subhadrabandhu (2001) on *A. champeden* seed is 0.5% – 1.5% (Table 4) while the crude fat in *A. heterophyllus* (jackfruit) seed was only 0.4%. Ripe *A. champeden* contained somewhat more crude fat than the unripe and hybrid species. The seeds had less crude fat than the flesh; the order of crude fat for seed samples was: ripe *A. champeden* > unripe *A. champeden* > *Nanchem*.

Total Carbohydrate

Ripe samples of fruit normally contain more carbohydrates than the unripe samples. Total carbohydrates (fresh cut) for ripe *A. champeden* was 16.2% – 28.3 g/100 g which was comparable to the reported value (25.8 g/100 g) (Janick &

Table 4. Proximate analysis of *A. champeden* (ripe and unripe) and *Nanchem* seed: Comparison of the present study with reported values on a dry weight basis (except ash and total carbohydrate).

Analysis (%)	Previous studies			Present study		
	A. champeden		*A. heterophyllus*	*A. champeden*		*Nanchem*
	Subhadrabandhu[b]	Zabidi & Aziz[b]	Haq[b]	Ripe	Unripe	
Moisture	46 – 78	56.57	64.5	52.9 – 72.8	83.6 – 91.1	49.7 – 54.0
Ash	3 – 4	2.57	–	3.2 – 5.1	4.0 – 5.0	2.8 – 3.2
Crude fibre	4 – 6	2.44	1.3	3.9 – 7.1	8.0 – 8.3	4.7 – 8.5
Crude protein	10 – 13	12.88	6.6	9.9 – 11.2	12.1 – 17.9	8.9 – 10.9
Crude fat	0.5 – 1.5	0.99	0.4	0.8 – 2.4	1.5 – 2.3	0.9 – 1.7
Total carbohydrate (g/100 g)[a]	77 – 81	24.55	25.8	2.8 – 3.5	1.8 – 2.5	3.2 – 3.4
Energy (kcal/100 g)	–	–	133 – 139	431 – 447	467 – 539	465 – 480

[a] Analysis carried out on fresh sample
[b] Values reported on dry weight basis

Paull 2008; Subhadrabandhu 2001) (Table 3) and was similar to *A. heterophyllus* (9.4% – 25.4 g/100 g) (Haq 2006; Janick & Paull 2008). The total carbohydrates for *Nanchem* (7.5% – 30.0 g/100 g) was similar to the reported values for *A. heterophyllus* (jackfruit) (9.4% – 25.4 g/100 g) (Haq 2006) and *A. champeden* (25.8 g/100 g) (Janick & Paull 2008; Subhadrabandhu 2001). Hence, *Nanchem*'s flesh contained more total carbohydrates than ripe *A. champeden*. The order of total carbohydrate for the flesh was: *Nanchem* > ripe *A. champeden* > unripe *A. champeden*.

The total carbohydrate for the seed on the other hand was very much lower than its flesh. Total carbohydrates in ripe *A. champeden* (2.8% – 3.5 g/100g) and *Nanchem* (3.2% – 3.4 g/100 g) were similar. Unripe *A. champeden* had the lowest total carbohydrate (1.8% – 2.5 g/100 g). However, the total carbohydrate of the seed in this study was not comparable with the values reported by Subhadrabandhu (2001), Zabidi and Aziz (2009) and Haq (2006) (Table 4) because it was done in fresh weight. As a result, the order of total carbohydrate for seed was: ripe *A. champeden* ≈ *Nanchem* ≈ unripe *A. champeden*.

Energy

Nanchem flesh (456 – 477 kcal/100 g) provided more energy than the *A. champeden*, whereas unripe *A. champeden* flesh had somewhat more energy than its ripe flesh. The energy of *A. champeden* (Janick & Paull 2008) and *A. heterophyllus* (Haq 2006; Janick & Paull 2008) flesh was 490 kcal/100 g and 301 kcal/100 g (Table 3), respectively. Therefore, ripe *A. champeden* and *Nanchem* energy content was slightly lower with the values reported by Janick & Paull (2008) on *A. champeden*. The reported value for *A. heterophyllus* (jackfruit) appeared to be unusually low. The order of energy content in flesh was: *Nanchem* ≈ unripe *A. champeden* > ripe *A. champeden*.

Unripe *A. champeden* seed (467 – 539 kcal/100 g) provided more energy when eaten compared to the ripe *A. champeden* (431 – 447 kcal/100 g) and *Nanchem* (465 – 480 kcal/100 g). However, the reported value for *A. heterophyllus* (jackfruit) seed is 133 – 139 kcal/100 g (Haq 2006) (Table 4) which is dramatically lower than the values obtained in this study. No values had been reported on the energy content of *A. champeden* seed.

Minerals

The mineral (nutrient) composition of *A. champeden* (ripe and unripe) and *Nanchem* were determined on its flesh and seed on a fresh weight basis (Table 5).

Potassium was the prevalent mineral followed by Mg, Mn, Ca, Zn, Na, Cu, Fe, Co and Ni respectively, in the flesh and seed. The amounts of potassium in flesh and seed are shown in Figure 7. Ripe *A. champeden* and *Nanchem* seed contained a higher amount of potassium than its flesh. This trend was however a reversed for the unripe *A. champeden*. The amount of potassium in ripe *A. heterophyllus* (jackfruit) is 292 mg/g (Janick & Paull 2008) (Table 3) which was lower than the amount reported in this study. On the other hand, the amount of potassium in unripe *A. champeden* flesh (288 mg/100 g) was similar to the unripe *A. heterophyllus* (287 – 323 mg/100 g) (Haq 2006).

The other major element present in the flesh and seed was magnesium (Figure 8). Similarly to potassium, ripe *A. champeden* and *Nanchem* seeds yielded a higher quantity of Mg than its flesh, while the reverse was seen between the unripe *A. champeden* flesh and seed.

Mn, Ca, Zn, Na, Cu, Fe, Co and Ni were treated as minor elements in this study because they are found at a concentration of less than

Table 5. Mineral content for *A. champeden* (ripe and unripe) and *Nanchem* flesh and seed (mg/100 g) on a fresh weight basis[a].

Mineral (mg/100 g)	Flesh			Seed		
	A. champeden		*Nanchem*	*A. champeden*		*Nanchem*
	Ripe	Unripe		Ripe	Unripe	
K	434 ± 38	288 ± 17	302 ± 53	609 ± 33	250 ± 61	594 ± 25
Mg	46 ± 6	41 ± 3	42 ± 7	65 ± 11	32 ± 6	87 ± 3
Mn	4.3 ± 1.0	5.2 ± 0.6	3.9 ± 0.2	5.5 ± 0.7	5.1 ± 1.1	0.7 ± 0.3
Ca	3.4 ± 1.5	5.1 ± 1.4	2.6 ± 0.5	2.9 ± 0.6	2.3 ± 0.3	3.1 ± 0.2
Zn	2.0 ± 0.0	1.9 ± 0.9	1.2 ± 0.0	1.9 ± 0.4	0.9 ± 0.3	1.1 ± 0.1
Na	1.1 ± 0.1	0.8 ± 0.1	1.6 ± 0.3	1.2 ± 0.2	0.8 ± 0.3	0.9 ± 0.1
Cu	1.1 ± 0.0	1.0 ± 0.2	1.0 ± 0.1	1.0 ± 0.0	0.7 ± 0.2	1.0 ± 0.0
Fe	0.5 ± 0.0	0.5 ± 0.0	0.4 ± 0.0	0.7 ± 0.1	0.6 ± 0.0	0.7 ± 0.0
Co	0.4 ± 0.0	0.3 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.2 ± 0.1	0.3 ± 0.0
Ni	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	0.2 ± 0.2
Cd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0
Pb		Not detected			Not detected	

[a] The mean of two replicates ± deviation

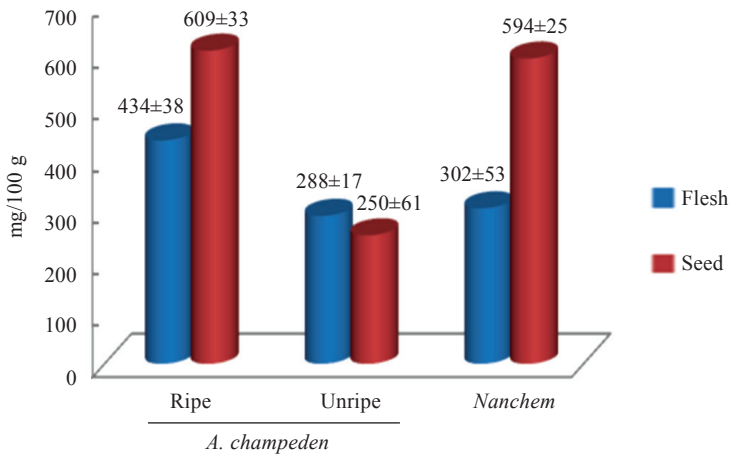

Figure 7. Potassium content in A. champeden *(ripe and unripe) and* Nanchem *flesh and seeds (mg/100 g), fresh weight basis, the mean of two replicates* ± *deviation.*

10 mg/100 g (Table 5), (Figure 9 and Figure 10). However, a small amount of cadmium was also detected in the flesh and seed samples, at about 0.1 mg/100 g. As suggested by FAO/WHO, a possible source of cadmium is due to soil contamination as cadmium could be easily absorbed by roots (Boisset & Narbonne 1995). Lead was opportunely not detected in any of the samples.

CONCLUSION

In terms of the flesh, unripe *A. champeden* was more nutritive than ripe *A. champeden* and its hybrid, *Nanchem*, in the area of ash, crude fiber and crude protein. In contrast, both ripe *A. champeden* and *Nanchem* had more total carbohydrates than unripe *A. champeden*. Unripe *A. champeden* is always eaten as a vegetable due to the lower sweetness. In terms

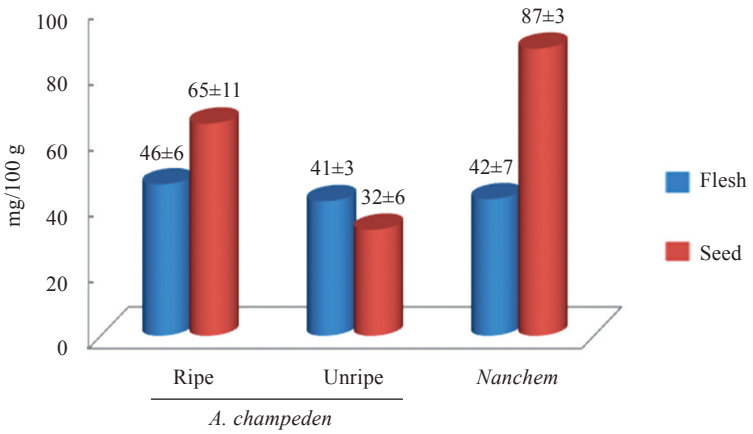

Figure 8. Magnesium content in A. champeden (ripe and unripe) and Nanchem flesh and seeds (mg/100 g), fresh weight basis.

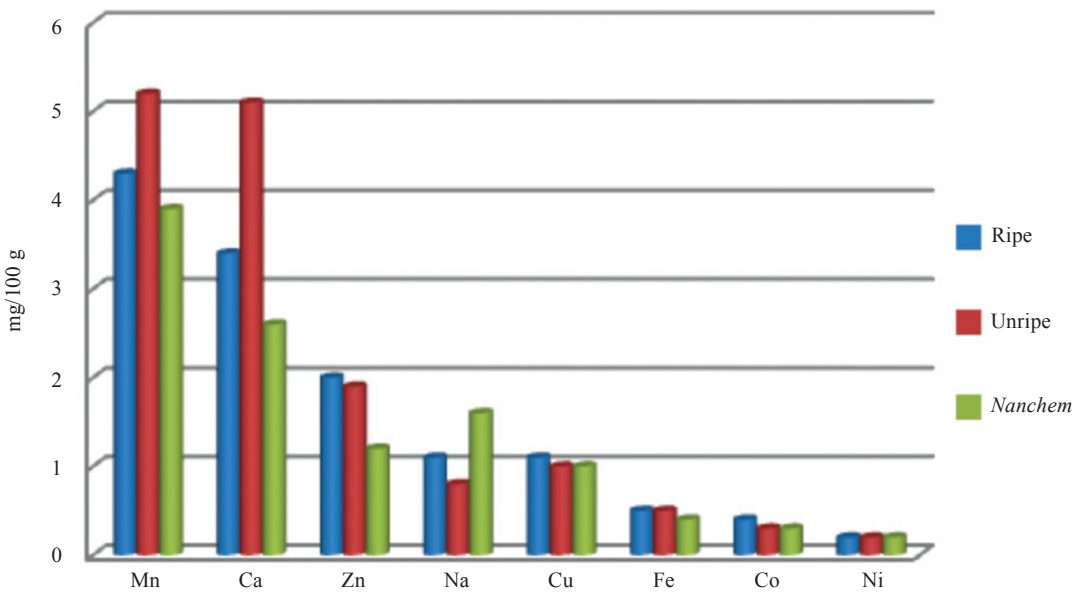

Figure 9. Minor elements present in A. champeden (ripe and unripe) and Nanchem flesh (mg/100 g), fresh weight basis.

of its high moisture content, it resembles a vegetable. The amount of sweetness in fruit is related to the total carbohydrates, but it also depends on its maturity, growing location and temperature (Kirk & Sawyer 1991).

Similar to the flesh, unripe *A. champeden* seed provided higher values for moisture, ash, crude fiber, crude protein, crude fat and energy than the ripe *A. champeden* and *Nanchem* seed. The similarities found between ripe *A. champeden* and *Nanchem* seed were moisture, crude protein, crude fat and total carbohydrate.

K and Mg were the prevalent minerals in the flesh and seed samples, while Mn, Ca, Cu, Fe, Co and Ni was present in quantities of less than 10 mg/100 g.

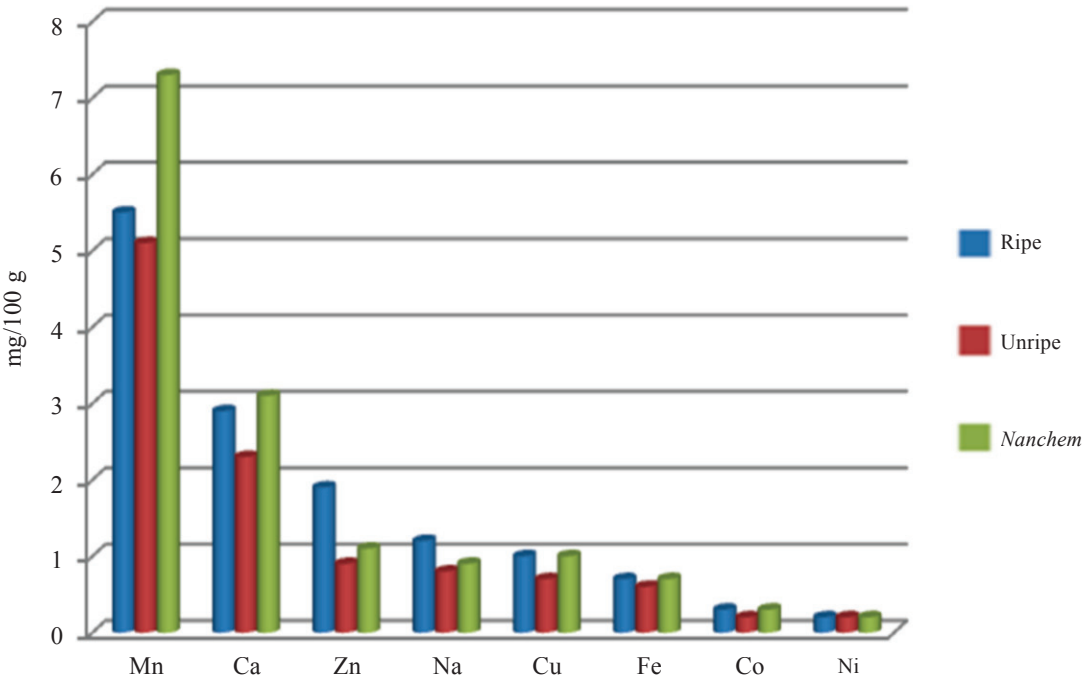

Figure 10 Minor elements present in A. champeden (ripe and unripe) and Nanchem seed (mg/100 g), fresh weight basis

The nutrient components in the flesh and seeds of *Nanchem* were closer to *A. champeden* in terms of moisture, ash, crude protein and crude fat, while *Nanchem* flesh resembled *A. heterophyllus* (jackfruit) in terms of ash and total carbohydrate. Therefore, it appeared that *Nanchem* had the characteristics of both *A. champeden* and *A. heterophyllus*.

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The authors would like to thank Universiti Brunei Darussalam and the Government of Brunei Darussalam for their financial support for this project and the Brunei Agriculture Research Center for the use of their equipment for crude fibre and protein analysis.

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Assessment of the Treatment of Textile Mill Effluent Using UASB Reactor

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This study was designed to evaluate the feasibility of the treatment of actual textile mill effluent using a upflow anaerobic sludge blanket (UASB) reactor. The main objective of this study was to generate design aids; in terms of organic loading rate (OLR), hydraulic retention time (HRT) versus chemical oxygen demand (COD) and colour removal in the textile effluent using a UASB reactor at neutral pH and constant mesophilic temperature. The COD, colour and total suspended solids concentration of the textile wastes used in the study were analyzed as 5440 mg/l, 3280 mg/l, 2320 units and 955 mg/l, respectively. The UASB reactor was started up by gradually increasing the OLR from 0.2 kg-COD/m^3-day to 2.6 kg-COD/m^3-day in order to prevent an organic shock to the reactor. Similarly, the hydraulic retention time (HRT) was slowly reduced from 58 h to 8 h to prevent the wash-out of sludge from the reactor. It was observed that more than 80% of COD and colour could be effectively removed at an OLR of 2.2 kg-m^3/d and HRT of 20 h. At optimum operating conditions, the effluent volatile fatty acid concentration was observed to be 430 mg/l. The average biogas production observed during this study was 0.34 l/g-$COD_{removed}$ and it was composed of 58% methane. During the course of maturity of granular sludge, its effective size and settling velocity were observed to increase exponentially as $0.261e^{0.051x}$ and $1.91e^{0.017x}$ respectively. The overall observed biomass yield (Y_{obs}) for the experimental period was calculated as 0.049 g-VSS/g-COD_{rem}. This study suggests that the use of a UASB reactor for textile mill effluent is a fairly feasible and viable option.

Key words: anaerobic digestion; textile mill effluent; neutral pH; COD; biogas; design parameters; constant temperature; OLR; HRT

Pakistan is facing acute water deficiency, its water resources have been declining day by day on account of diversified environmental and erroneous water management issues. The available water is being contaminated at a deplorable rate owing to the dumping of untreated domestic and industrial effluent together with agricultural/surface runoff into the receiving waterways. Clean drinking water is therefore only accessible to 18% of its population and the rest of the population are getting polluted drinking water, as designated by the required WHO standards (PCRWR 2005). Moreover, it is an admitted truth that Pakistan has a deficient energy sector despite the fact that it has ample potential for its production. The energy supply deficiency also applies to the electricity sector. Bio-gas is however playing a key role in this sector, which is approximately equal to 37% of the total energy production. Since, it's the most cost-effective and environmentally friendly source of energy, it requires more awareness and consideration for it to undertake the resolution of the energy crises

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by the latest developed techniques of bio-gas generation, especially by converting wastes into energy (Arshad & Hashim 2010). As a result of this, not only the energy dilemma could be worked out but the problem of disposing the untreated domestic and industrial wastes can also be resolved.

At present, there are numerous textile mills contributing significantly to the country's Gross Domestic Production (GDP) as the backbone of its economy. Textile manufacturing is however, one of the most polluting industries, it liberates heavily polluted effluent. Approximately 600 m^3 of wastewater is discharged from the textile mills for each kg of textile production. Wastewater contains more than 1000 mg/l of chemical oxygen demand (COD) in addition to a variety of dyes that impart unaesthetic colour to the receiving water courses (Pak-EPA 1999). The release of coloured compounds into the environment is extremely obnoxious because they destroy aquatic life by affecting photosynthesis; moreover, several dyes and their breakdown products are toxic and mutagenic to life (Somasiri *et al.* 2010; Paulo Igor *et al.* 2010). Consequently, textile waste management is an essential operation towards protection of the environment.

Although the available physical and chemical wastewater treatment techniques are fairly effective, they become expensive when applied on a larger field scale. Therefore, biological treatment techniques are considered as viable options for waste management, especially for developing nations (Bhatti *et al.* 1996; Savant *et al.* 2005; Arshad *et al.* 2008). The biological treatment methods are primarily of two kinds, aerobic process and anaerobic process. Due to the larger energy and nutrition input requirements of the aerobic process, comparatively more importance is given to anaerobic digestion. The utilization of anaerobic digestion which is fairly cheaper in operation, is certainly one of the most excellent wastewater treatment alternatives left for the various types of domestic and industrial effluent all over the world (Lettinga *et al.* 1980; Schellinkhout 1993). The basis of its recognition around the globe is its simple procedure, technical feasibility, cheaper running and maintenance cost, proficient treatment of high-strength wastes etc. The treatability of actual textile waste at a mesophilic temperature range using the UASB reactor still seems to be unconvincing and doubtful, as the available studies do not precisely indicate the effects of organic loading rate (OLR) and hydraulic retention time (HRT) on the COD and colour removal efficiency of the reactor (Isolina *et al.* 2010; Kaan *et al.* 2009; Sreekanth *et al.* 2009; Sarayu *et al.* 2009; Ilter *et al.* 2010).

Since the upflow anaerobic sludge blanket (UASB) reactor is the most common type of anaerobic digestion generally employed for industrial effluent treatment (Arshad *et al*. 2009), this study was designed using a single-stage UASB reactor to investigate the treatment feasibility of textile wastewater under anaerobic conditions. The main objective of this study was to investigate the effects of design parameters like OLR and HRT on the treatability performance of the reactor under steady state conditions of neutral pH and constant temperature.

MATERIALS AND METHODOLOGY

Experimental UASB Reactor

Owing to prior knowledge and the ample advantages of the UASB reactor, it was decided to utilize a UASB reactor for the study. The reactor was fabricated using acryl resin with a total effective volume of 4.5 l, together with a thermostatic casing to maintain steady temperature inside the reactor. The reactor was also equipped with a gas solid separator (GSS) and a mixing apparatus (turbine shaped, 3.81 cm × 7.62 cm) to facilitate the appropriate confluence of substrate and biomass (Arshad & Hashim 2010). A methodical illustration of the UASB reactor is shown in Figure 1.

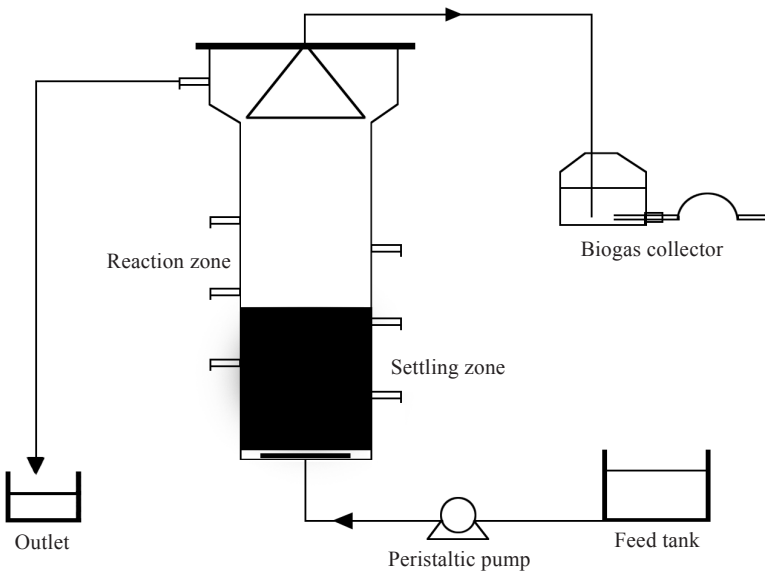

Figure 1. Methodological illustration of the UASB reactor.

Substrate and Nutrients

Actual effluent from the nearby textile mills was used in the study. Nitrogen and Phosphorous were added in the form of $(NH_4)_2SO_4$ and KH_2PO_4 in accordance with the COD:N:P ratio of 550:5:1. The stock solution of trace nutrients containing ferric chloride, zinc sulphate, copper sulphate etc. was added at a concentration of 1.0 milliliter per litre of the feed solution to the reactor (Bhatt 1995; Arshad *et al.* 2009). The wastewater characteristics of the textile mill used in the study is shown in Table 1.

Seed Sludge

About eighty percent volume of the reactor was filled with seeded sludge, which had been fully acclimatized with the substrate during the initial 20–22 days of typical laboratory procedures (Littinga *et al.* 1984; Arshad *et al.* 2010). The characteristics of the seeded sludge at the start up of the reactor are shown in Table 2. The seeded sludge provided 42 grams of volatile suspended solids (VSS). The loading rate was increased stepwise in order to avoid organic loading shock. HRT was also monitored and observed. Mixing was done twice a day.

Operational Conditions

Temperature. The temperature was kept constant at 28°C–30°C during the course of the study period, almost analogous to that of the actual effluent of the textile mill.

pH. A neutral pH was sustained by adding an external buffer solution ($NaHCO_3$) to the feed solution of the reactor.

OLR/HRT. The reactor was operated stepwise by gradually increasing the organic loading rates (OLR); starting from 0.2 kg-COD/m^3-day and getting it to the highest limit of 2.6 kg-COD/m^3-day during the course of study period. The HRT was however slowly reduced from 58 h to 8 h.

Monitoring and Analysis

pH, temperature, influent and effluent COD, volatile fatty acid (VFA) and gas production were monitored regularly, twice a week. Gas was collected over tap water saturated with NaCl. All analysis was carried out in accordance with standard laboratory techniques (AWWA 2005).

Table 1. Wastewater characteristics of the textile mill.

Parameters	Concentration
pH	8.5–9.1
Colour (units)	2320
COD (mg/l)	5440
BOD (mg/l)	3280
Total solids (mg/l)	2885
Total dissolved solids (mg/l)	1450
Total suspended solids (mg/l)	955
Total volatile solids (mg/l)	485

Table 2. Characteristics of seeded sludge.

Parameters	Value
Total solids (mg/l)	63.5
Total suspended solids (mg/l)	51.84
Volatile suspended solids (mg/l)	29.00
Colour	Blackish

RESULTS AND DISCUSSION

Start-Up of the Reactor

Since, pH and temperature are the most significant governing parameters for anaerobic digestion, extreme care was made during the course of study period to maintain them at an optimum level. Past experiences and literature reviews illustrate that a mesophilic range of temperature is the most suitable range for anaerobic conditions, as lower temperature inhibits microbial activities, while elevated (thermophilic) temperature gives rise to high endogenous death of microbes (Buhr *et al.* 1977; Switzenbaum *et al.* 1980; Kennedy *et al.* 1982; Henze *et al.* 1983; Grin *et al.* 1985). Therefore, the temperature was kept constant at a mesophilic range by placing water heating jackets around the reactor. The average temperature recorded during the course of the study period was 28°C–30°C. Likewise, neutral pH is considered to be the most appropriate range for anaerobic digestion (Bhatti *et al*. 1996). Consequently it was kept constantly at a neutral range, to attain maximum benefit from the process, by adding an external buffer solution of 0.03 M $NaHCO_3$, at the rate of 60 ml per litre, to the feed solution of the reactor (Arshad & Hashim 2010). The time course of pH during the study period is shown in Figure 2.

The reactor was started up in accordance to standard procedure and guidelines. The OLR was slowly raised from 0.2 kg-COD/m^3-d to 2.5 kg-COD/m^3-d and the HRT was gradually reduced from 2.8 d to 8 h to avoid any organic distress to the reactor. The treatability performance was also assessed during the course of the study period by observation of the concentration of VFA in the effluent of the reactor. VFA is one of the critical parameters which indicate the failure of the reactor to recover methane from the acetic acid. If the concentration of VFA exceeded 500 mg/l, the feed should be temporarily stopped to prevent further accumulation within the reactor or they could also be diluted by draining out part of the reactor fluid and adding some tap water (Riera *et al.* 1985; Mahadevaswamy *et al.* 2004). During this study, the average concentration of VFAs was observed to be 428 mg/l while its maximum value was observed to be 810 mg/l when the pH

of the reactor dropped drastically to the acidic medium. The time course of VFA during the study period is shown in Figure 3.

Treatability Performance

During the course of the study period, the overall COD and colour removal efficiency of the reactor under varying OLR and HRT were carefully observed. Increases in the COD and colour removal efficiency were observed during every step of steady state conditions of the reactor under a given OLR or HRT. Previously, it had been reported that the HRT did not significantly affect decolourization especially from 24 h to 12 h (Paulo Igor *et al*. 2010) but during this study a sudden decrease in the reactor's efficiency was observed for every step of either increasing the OLR or decreasing the HRT. This might be due to the accumulation of

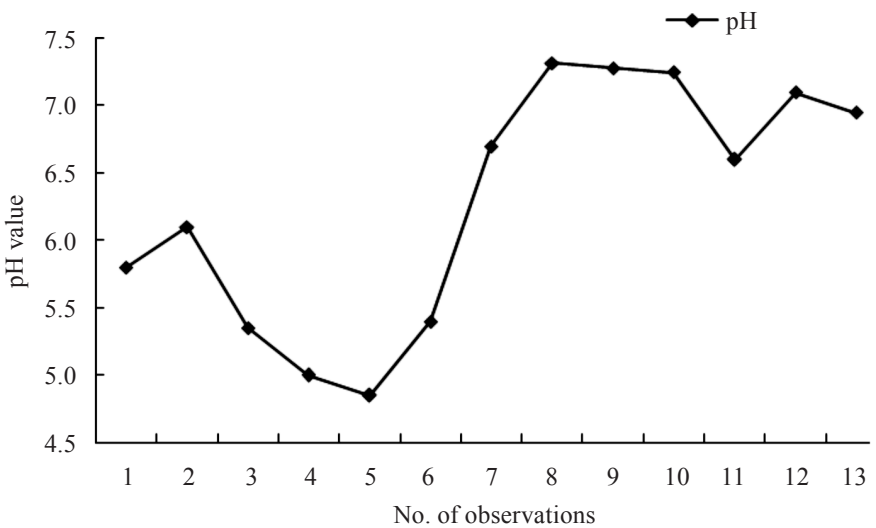

Figure 2. Time course of pH during the study period.

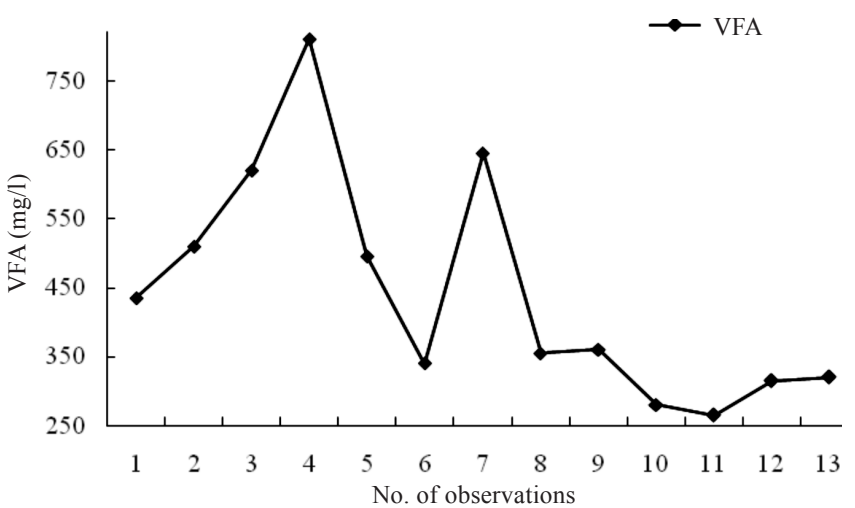

Figure 3. Time course of VFAs during the study period.

excessive VFA in the reactor at these operating conditions. The effects of OLR and HRT on the COD and colour removal efficiency of the reactor are illustrated in Figures 4–7.

The results acquired illustrate that lower OLR and higher HRT favour the treatability performance of the reactor. It was observed that corresponding to optimum conditions, i.e. OLR of 2.2 kg-m^3/d and HRT of 20 h, the COD and colour removal efficiency of the reactor were more than 82% and 79%, respectively. Almost comparable treatability performance has been reported earlier while working on the textile mills effluent under anaerobic conditions (Somasiri *et al.* 2010; Wijetunga *et al.* 2008). In fact, the data acquired from this study presents practical guidelines for the design of a UASB reactor for textile mill effluent at various OLRs and HRTs.

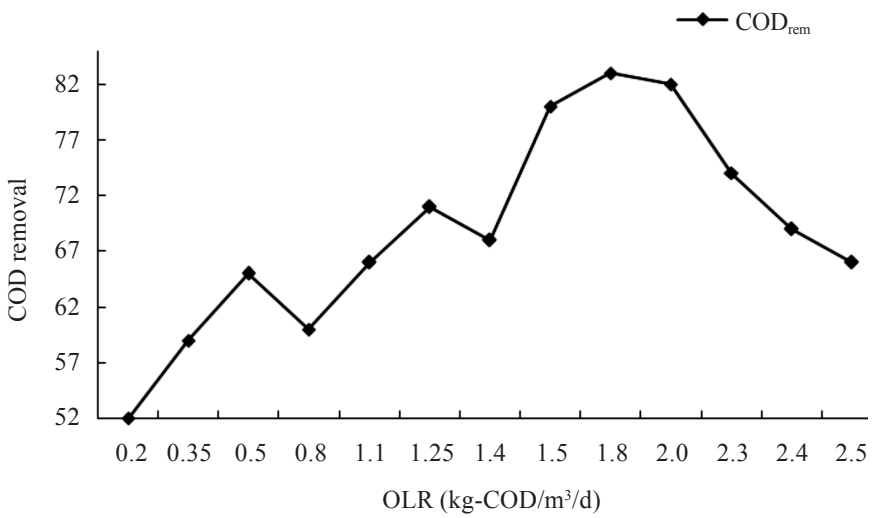

Figure 4. Effects of OLR on COD removal.

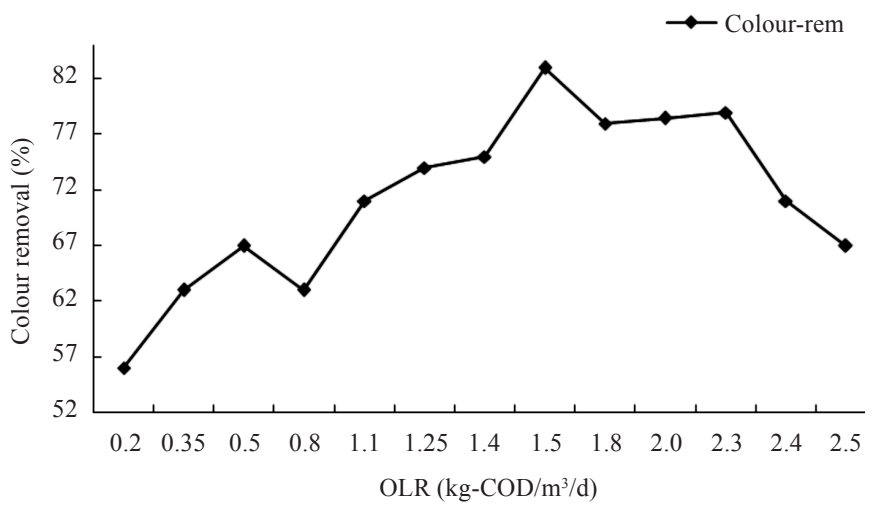

Figure 5. Effects of OLR on colour removal.

Treatment assessment of different studies conducted on various types of waste using a UASB reactor is shown in Table 3. This evaluation demonstrates the effectiveness of a UASB reactor for the treatment of textile wastes analogous to the pulp and paper mill wastes, sugary wastes etc. The slightly lower treatment efficiency of the UASB reactor for the textile mill effluent, as was also evident from this study, might be due to the existence of a high proportion of material which were not susceptible to anaerobic digestion in the wastes that were used.

Bio-gas Production

Figures 8 and 9 illustrate the amount of biogas production and the methane composition observed during the course of the study period. It was noticed that the amount of biogas production varied linearly with the HRT. The higher the HRT, the more would be the biogas

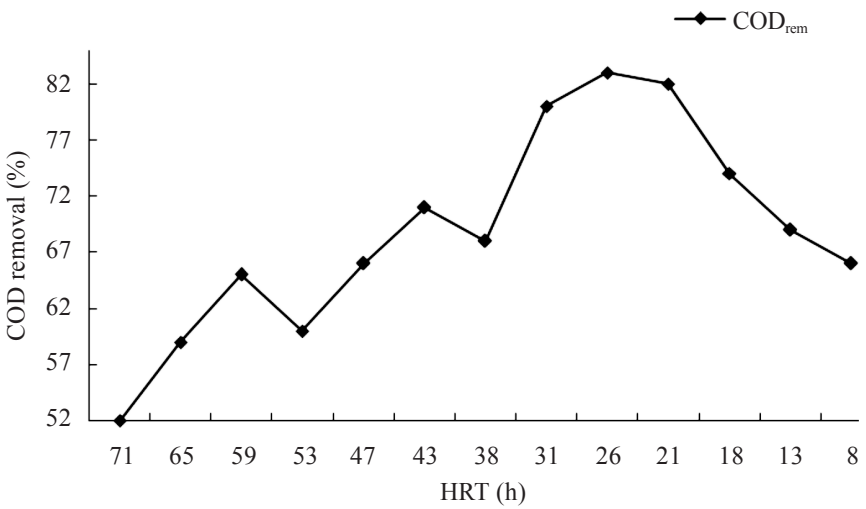

Figure 6. Effects of HRT on COD removal.

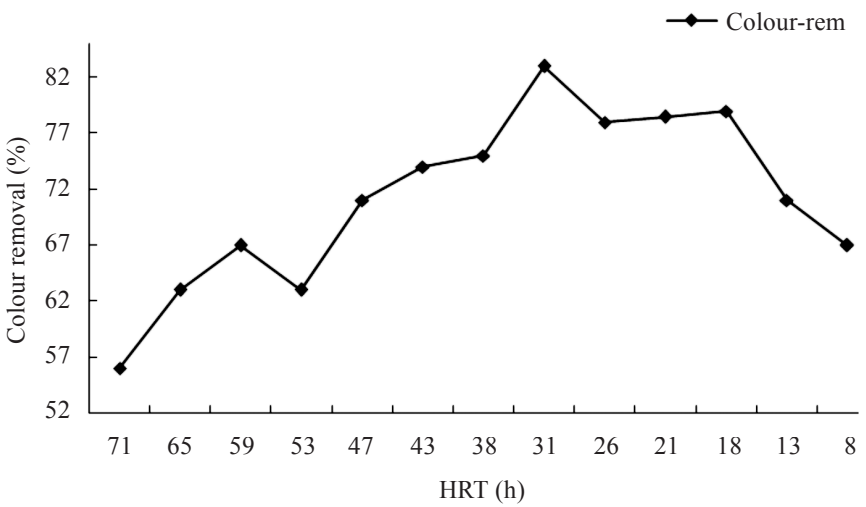

Figure 7. Effects of HRT on colour removal.

Table 3. Treatment assessment of the UASB reactor using various types of waste.

Substrate	Removal efficiency	ORL/HRT	Reference
Domestic effluent	COD = 85%	OLR = 1.8 kg-COD/m^3-d	Arshad *et al*. 2008
NSSC pulping effluent	Lignin-COD = 38%	OLR = 2.75 kg-COD/m^3-d HRT = 38 h	Arshad *et al*. 2009
Paper mill effluent	COD = 64%	OLR = 2.14 kg-COD/m^3-d	Arshad & Hashim 2010
Sugar mill effluent	COD = 70%	OLR = 2.1 kg-COD/m^3-d HRT = 16 h	Arshad *et al*. 2010
Textile mill effluent	COD = 82% Colour = 79%	OLR = 2.2 kg-COD/m^3-d HRT = 20 h	This study

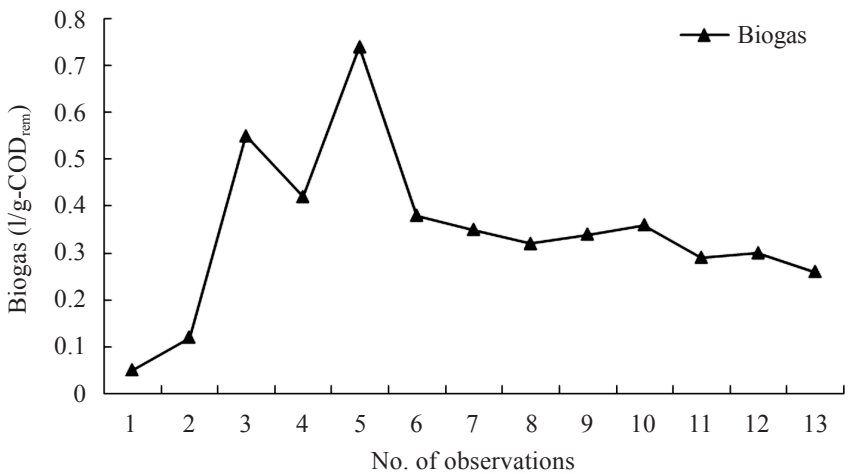

Figure 8. Amount of biogas production.

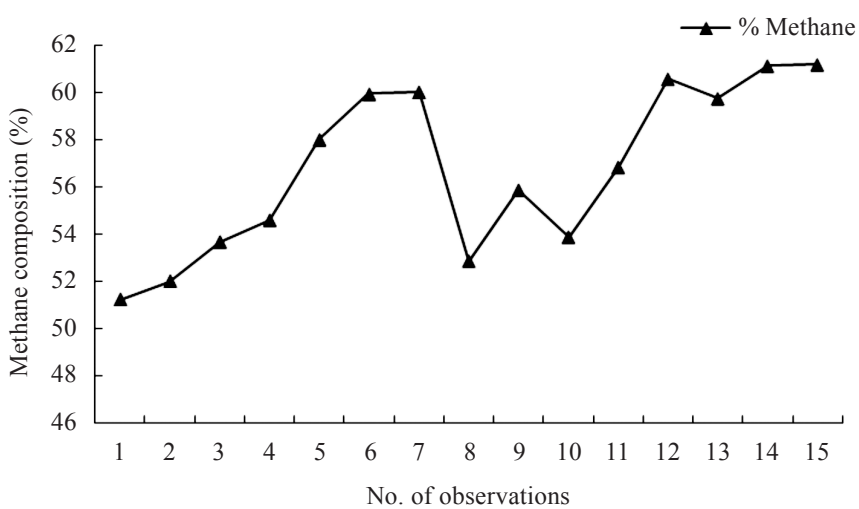

Figure 9. Methane composition of the biogas.

production and vice-versa; and that might be due to the fact that the lower HRT promoted the wash-out of sludge from the reactor, which ultimately affected the biogas generation. In addition, the low mixing of the biomass and the substrate also reduced the biogas production.

The main constituents of the biogas as reported earlier were methane and carbon dioxide that were produced during anaerobic digestion (Bhatti *et al*. 1996; Mahadevaswamy *et al.* 2004). A small proportion of the biogas was H_2, if the reactor was not functioning properly, due to the existence of hydrogen-producing acetogens that provided adverse operational conditions for the conversion of VFA to acetate rather then to methane.

The average gas production observed during the study was 0.34 l/g-$COD_{removed}$ that nearly resembled the theoretical value of 0.35 l/g-$COD_{removed}$ (Arshad *et al*. 2010), but the percentage composition of methane was comparatively low, i.e. 57.88% (Bhatti 1995, Arshad *et al.* 2009) which indicated the presence of recalcitrant substances in the textile waste. As the granules had not matured during the initial study period, the amount of biogas observed during that stage was comparatively lower, i.e. 0.08 l/g-COD_{rem}-d composed 52% of methane.

Sludge Characteristics

The sludge character is an important factor in evaluating the treatability performance of the reactor. The seeded granular sludge that was dark brownish in colour at the start-up period became grayish-dark in colour after a few weeks of the process. The effective size and settling velocity of granular sludge during the course of the study period is shown in Figures 10 and 11, respectively. The figures illustrate that the effective size and settling velocity of the granular sludge gradually increased during the course of digestion until they were entirely mature. The equations obtained from the data are shown below:

Granular size development

$$y = 0.261e^{0.051x} \quad (1)$$

Settling velocity improvement

$$y = 1.91e^{0.017x} \quad (2)$$

At the initial period of the study, the enhancement in the effective size of sludge was comparatively lower, which was mainly due to the wash-out of sludge from the reactor. The effective size of granular sludge previously reported varied from 0.3 mm–5 mm for different types of waste (Pol 1983; Mahadevazwamy *et al.* 2004). In this study, the average effective size and settling velocity of the granular sludge were observed to be 0.38 mm and 2.17 cm/s, respectively. The smaller granule size observed was mainly due to the shorter period of the study and the employment of an insignificant OLR.

The mean ash content of the granular sludge was observed as 10.12%, which decreased to 8.84% at the end of study period. The MLSS concentration and the VSS/MLSS ratio of the granular sludge were recorded at 56 400 mg/l and 0.81 mg/l, respectively. The overall observed biomass yield (Y_{obs}) for the experimental period was calculated as 0.049 g-VSS/g-COD_{rem} by using the following equation:

$$Y_{obs} = \sum X / \sum S \quad (3)$$

Where,
$\sum X$ = total biomass produced, g-VSS and
$\sum S$ = Total substrate removed, g-COD

This value of Y_{obs} was almost one-tenth of the typical activated sludge process (Bhatti *et al.* 1996). Thus, it was consistently shown by means of this study that anaerobic digestion produced comparatively less sludge than the aerobic process.

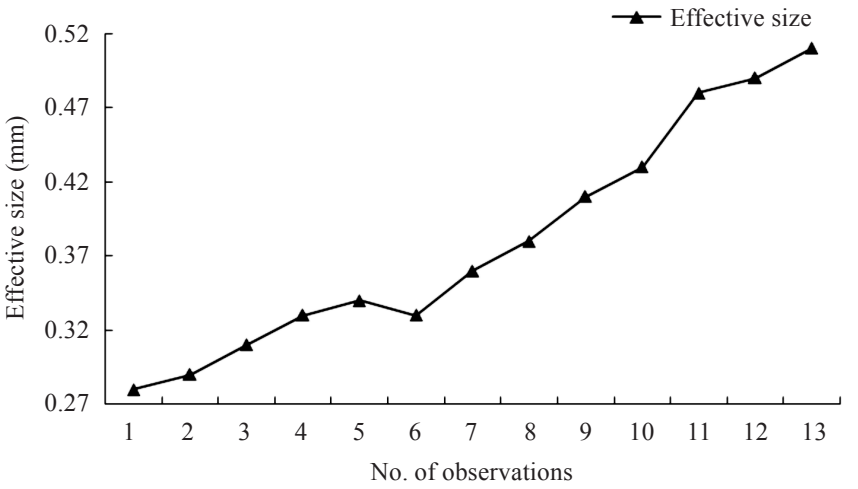

Figure 10. Effective size of the granular sludge.

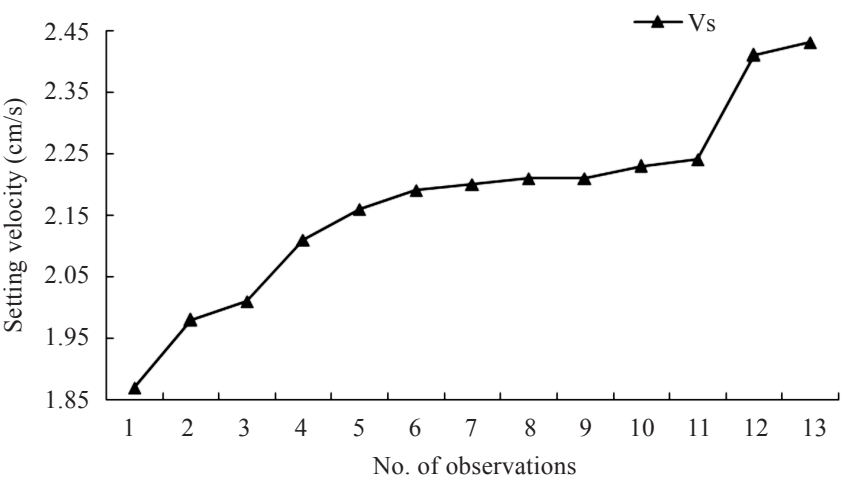

Figure 11. Settling velocity of the granular sludge.

CONCLUSION AND RECOMMENDATION

From the results of this study, the following conclusions were drawn:

- The textile industry's waste management was a serious environmental concern that should be tackled appropriately. Due to the limited energy and resources crises being faced by the developing world, anaerobic digestion was one of the best options available for the precise management of various kinds of waste including effluent from textile mills.
- The wastewater being released from textile industries, which was of high-strength, alkaline and coloured in nature, was quite feasible for anaerobic digestion. The COD, BOD, TDS, TSS and colour concentration values of the textile mill effluent were

5440 mg/l, 3280 mg/l, 1450 mg/l, 955 mg/l and 2320 units, respectively.

- More than 80% of the COD and colour could be removed from textile effluent at an OLR of 2.2 kg-m^3/d and HRT of 20 h.
- Sustaining neutral pH by adding an external buffer solution $NaHCO_3$ to the reactor, the VFA of the effluent concentration could possibly be kept at less than 430 mg/l.
- An average gas production of 0.34 l/g-$COD_{removed}$ could be obtained from the textile mill effluent by using a UASB reactor at constant neutral pH and mesophilic temperature.
- The lower methane composition of the biogas generated during the study, i.e. 57.88% reveal the presence of recalcitrant substances in the textile mill effluent.
- The effective size and settling velocity of the granular sludge enhance exponentially through their development, i.e. $0.261e^{x0.051}$ and $1.91e^{x0.017}$, respectively.
- The mean ash content, MLSS concentration, VSS/MLSS ratio of the granular sludge was 10.12%, 56 400 mg/l and 0.81 respectively, while the overall biomass yield (Y_{obs}) was calculated as 0.049 g-VSS/g-COD_{rem}.

Treatability of textile effluent in a single-stage UASB reactor at neutral pH and mesophilic temperature is a highly feasible, cost-effective and reliable technique. A long-term comprehensive study is required to study the exact behaviour of textile effluent under anaerobic conditions using variable operating conditions. The nature and concentration of recalcitrant material in the textile effluent and their overall impact on anaerobic digestion needs to be evaluated.

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Field and Laboratory-based Approach for the Determination of Friction Angle of Geological Discontinuities of Malaysian Granites

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The basic friction angle, Φ_b for artificially sawn discontinuity planes for fresh granite, as determined by tilt testing, has an average value of 30º. For the natural rough discontinuity surfaces, a wide range of values have been determined for the peak friction angle, Φ_{peak} ranging from 47º to a maximum value of 80º, depending on the joint roughness coefficient (JRC). The average values of the friction angles for the different degrees of roughness were as follows: JRC 2–4 = 58°; JRC 6–8 = 60°; JRC 8–10 = 47°; JRC 12–14 = 60°; JRC 14–16 = 71° ; JRC 18–20 = 80°.

Key words: tilt testing; peak friction angle; roughness coefficient; stability; rock masses; comparison; estimation

The surface roughness and frictional properties of geological discontinuity surfaces play an important role in assessment of the stability of rock masses, for example in underground excavation and in particular for cut rock slope stability assessment. This surface roughness can be expressed as the coefficient of friction or the friction angle of the rough discontinuity surface. However quantification of this parameter has generally been neglected in Malaysia and the results presented here are the findings of a recently conducted systematic study of this parameter. While sophisticated and costly, laboratory machines involving complicated testing procedures exist for determination of the shear strength of discontinuities from which the friction angle can be determined. The approach adopted in this research was to apply relatively low cost and easy applicable methods to quantify the friction angle of the discontinuity plane surfaces for the findings to attain wider applications. Hoek (2007) with over forty years experience in rock mechanics, recently recommended this approach because a larger number of tests can be conducted; mean values and their range can also be determined, and the interpretation of the results can better represent the site conditions. The tilt test has been employed to determine the angle of friction of both natural as well as artificially prepared sawn discontinuity surfaces for fresh to slightly weathered granite. The testing procedures closely follow the recommendations of Priest (1993). The joint roughness coefficient (JRC) as defined by Barton and Choubey (1977) is measured in the field as well as in the laboratory. A comparison of these two findings presents the possibility of estimation of the friction angle of the discontinuity plane surfaces based on JRC which can be rapidly determined during a field survey.

MATERIALS AND METHODOLOGY

Basically two approaches have been applied for the determination of friction properties of the

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discontinuity surfaces: a field and laboratory determination of JRC as defined by Barton and Choubey (1977); and tilt testing of both natural and artificially prepared sawn discontinuities in accordance to the procedures outlined by Priest (1993). Fresh and slightly weathered granite rock blocks collected from the different sites were cut into approximately 6 cm cubic pieces which were then sawn into two pieces to obtain the artificial discontinuity surfaces. In addition, at each site a systematic discontinuity survey was conducted to characterise the rock mass. In this survey, the surface roughness of the naturally occurring discontinuity surfaces was determined using a profiler such as the one shown in Figure 1.

During the field survey, matched pairs of rock material blocks containing natural discontinuities with different JRC values were also collected. Both the sawn pieces as well as the naturally occurring discontinuities with different degrees of roughness were subjected to tilt testing using the self-fabricated simple testing apparatus shown in Figure 2. In this manner, both the basic friction angle, Φ_b obtained for the artificially sawn surfaces, as well as the peak friction angle, Φ_{peak} for the natural surfaces with different degrees of roughness were determined.

For this investigation, field surveys and sampling were conducted at four different sites underlain by granite as follows:

- Kajang Rock Quarry, Kajang, labelled as KR,
- SILK Highway, Kajang, labelled as SH,
- Pos Selim to Kg.Raja road, labelled as PS; and
- Bukit Penggorak Quarry, Kuantan, labelled as BP.

Figure 3 shows the locations of the investigated sites.

RESULTS AND DISCUSSION

A summary of the results of tilt testing of artificial and natural discontinuities as well as the JRC values for the investigated granite discontinuities are presented in Table 1.

The following points can be summarised from the results of this investigation:

- For the sawn surfaces, the artificially prepared discontinuities, the friction angle for both the fresh as well as the slightly weathered surfaces were within the same range of overlapping values. This result indicated that slight weathering did not cause a

Figure 1. Profiler employed for the determination of JRC.

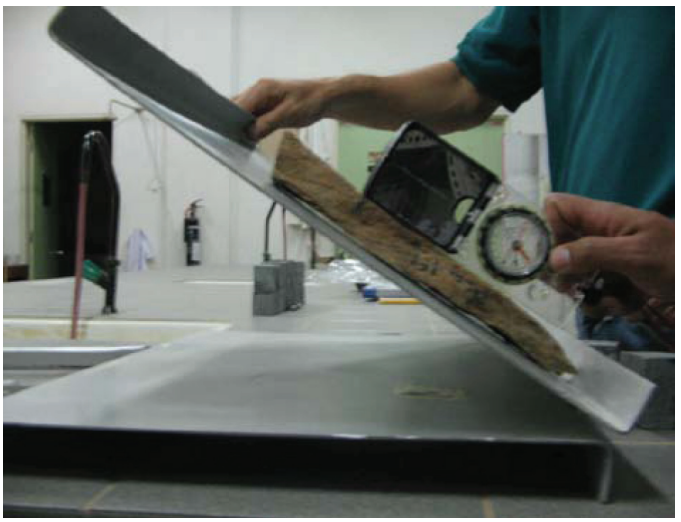

Figure 2. Simple tilt test apparatus for determination of peak friction angle, Φ_{peak} of naturally occurring discontinuities.

Table 1. Mean values and range for friction angles of geological discontinuities of investigated granites.

Discontinuity roughness	Sawn surface (Fresh)	Sawn surface (Slightly weathered)	JRC 2 – 4	JRC 6 – 8	JRC 8 – 10	JRC 12 – 14	JRC 14 – 16	JRC 18 – 20
Mean angle [°]	30.10	29.90	58.30	59.70	47.30	59.70	70.80	79.90
Range [°]	29.0–32.0	28.5–31.5	48.0–66.0	52.0–67.0	29.0–63.0	59.0–60.0	64.0–76.0	73.0–88.0

reduction in the surface roughness of these surfaces. In other words, slight weathering of the rock did not cause sufficient deterioration of the rock material surface to result in a decrease in its surface roughness.

- Generally stated, discontinuities with higher JRC values had higher friction angles. However, for a given JRC value, the range of friction angle values from tilt testing could have a wide range of up to ten degrees.
- Within a given granitic rock mass, for example Kajang Rock Quarry, different JRC values where determined for different discontinuity sets. In the Kajang Rock Quarry for example, for the different discontinuities sets, the JRC values of 2–4, 6–8, 8–10,12–14 and 18–20 were determined. This finding indicated the importance of the inclusion of JRC measurement during field discontinuity surveys and the determination of JRC for specific discontinuity sets.

From Table 1, the basic friction angle, Φ_b for the tested granites was determined as 30°. Depending on the degree of roughness, the peak friction angle, Φ_{peak} attained a maximum value of 80° depending on the JRC value. These findings are in general agreement with studies by other researchers, for example Barton (1976), Barton and Choubey (1977) and Hoek and Bray (1981). This study also illustrates the

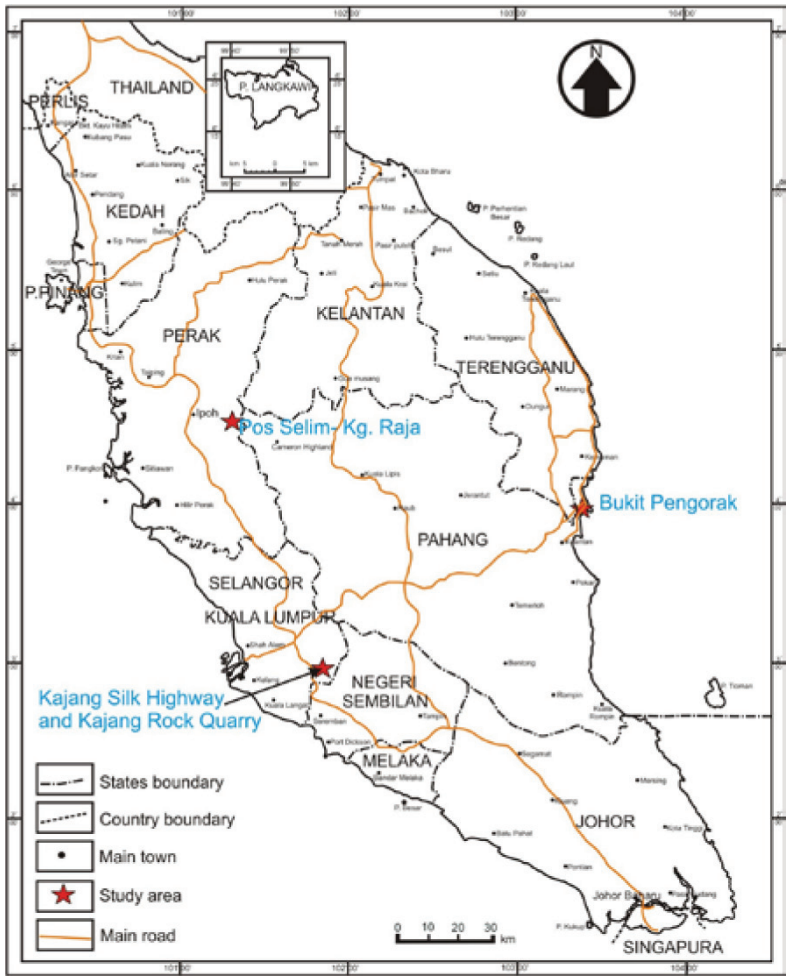

Figure 3 Location of investigated sites.

importance of the determination of JRC values as a means of estimating the friction angle of discontinuity surfaces in the field based on the establishment of a qualitative correlation such as the one presented in this study (Table 1). Similar studies are currently being undertaken for the other major lithologies in Malaysia.

CONCLUSION

The basic friction angle Φ_b for granite was determined as 30° and the peak friction angle Φ_{peak} varied from 47° to as high as 80° depending on the roughness of the discontinuity surface as shown by its JRC. Determination of JRC in the field could be employed to estimate the friction angle of the discontinuity plane surfaces for the granitic rock masses.

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Recycling Mimeograph-Printed Newsprint Paper

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Virgin newsprint paper from thermo-mechanical pulp was subjected to a laboratory recycling scheme, which involved mimeograph-printing, re-pulping, de-inking, washing, refining, and handsheet-forming, without adding other fibre in between cycles. Fibre dimension, pulp freeness and paper properties were determined after each cycle until the fifth, at which about 20% of the original material remained. The remaining fibre was then mixed with virgin pulp, the original newsprint and unsorted mixed office waste to determine the proportion necessary for acceptable properties.

The results indicated remarkable modification in distribution of fibre properties, a decreasing amount of long fibre with corresponding increase of short fibre in the course of recycling and loss of fibre. Refining generally improved the strength properties of paper from recycled fibre.

About 20 % to 30 % of either thermo-mechanical pulp or unsorted mixed office waste was found sufficient for blending with recycled fibre to obtain acceptable strength properties.

Key words: tmp newsprint, handsheet, number of recycling, paper properties

History says that papermaking actually started in China about 2000 years ago from recycled waste material of rotten fishnets. Later, recycling of fibre or waste paper was also developed. Proof of its development as early as the l4th century can be found in Venice, Italy.

There are many benefits claimed from recycling paper, namely, (Manfred 1985) preserves forests — approximately seventeen trees are saved for every ton of paper recycled; (2) conserves water — recycled paper uses 55% less water in its manufacture than new paper; (3) reduces water pollution by 35%, reduces air pollution by 74% and eliminates many toxic pollutants; (4) saves more than 3.3 cubic yards of landfill space per ton of recycled paper and (5) saves 60% – 70 % of energy used for virgin pulp (CencalRecycling, 2011).

Recycling of papers, admittedly, is beneficial in several aspects and the process continues to be developed as research continues on what happens to the fibre as it undergoes recycling. A review of literature by Howard (Sutjipto *et al*. 2008) illustrated that recycled pulp quality and recycled paper properties depended only partly on the recycling process itself but rested mainly on the 'history' of the material being recycled and on the processes and treatments experienced by the material during papermaking, converting and use. Chemical pulp, unrefined chemical pulp, and mechanical pulp appear to behave differently during recycling. Although extensive research has led to the identification of many of the effects of the relevant factors, the review concluded that there were still gaps to be worked on to understand the implications of using higher levels of recycled fibre.

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A recent study dealt on the effect of recycling on properties of paper from different sources of pulp (Howard 1993). Reports on recycling, however, have dealt mainly on re-slushing, refining and handsheet-forming, and hardly include the actual printing on the material being recycled. This particular paper reports a recycling scheme using virgin newsprint paper and a mimeographing machine for printing. Mimeographs are well-known duplicating machines used in schools and offices for the reproduction of instructional material and office forms. The recycling was also planned such that no other fibre was added in-between cycles until the fifth cycle.

The study was conducted to determine (1) the changes in the properties of virgin newsprint paper and its fibre due to recycling and (2) the effects of blending the recycled fibre with virgin pulp, mixed office waste or virgin newsprint paper.

MATERIAL AND METHODS

Material

The main material used was virgin newsprint paper from thermo-mechanical pulp (TMPN) obtained from Norway. Virgin pulp (TMP) was also requested for the blending experiment. Unsorted mixed office waste paper (UMOW), also used in the blending experiment, was collected from the FPRDI offices.

The ink used for printing was ordinary mimeographing black ink. The chemicals used for deinking and the corresponding levels of addition were as follows (Basis: ODW pulp):

Chemicals	Charge (%)
Sodium hydroxide	1
Hydrogen peroxide	1
Dispersant	1
Foamer/collector	3
Sodium silicate	1
DTPA (Diethylenetriamine pentaacetic acid)	0.2

METHODS

Recycling Process

One recycling cycle involved printing, disintegration or re-pulping, de-inking and flotation, washing, refining, and handsheet forming (Figure 1). No other fibre were added in-between cycles until the fifth cycle.

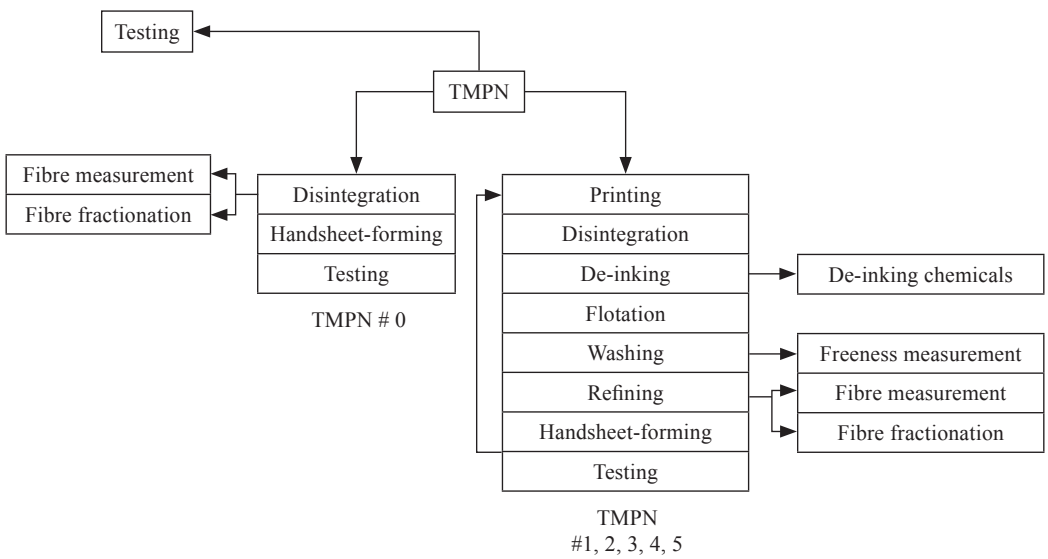

Figure 1. Laboratory recycling scheme.

The raw material TMPN was used as a printing substrate for mimeographing. The printed raw material was then manually shredded and repulped initially in the laboratory disintegrator at a 4% consistency level (using water at 45°C – 50°C) for 4 min. The repulping/de-inking chemicals were then introduced into the slurry for a total retention time of 15 min. The pH of the slurry was within 10.7 – 11.5. The slurry was then transferred into the flotation cell. The consistency was lowered to 1% by adding water. Compressed air was introduced into the cell. This generated bubbles in the slurry, which carried the ink particles after being skimmed off the cell. This stage was done within 10 min. The pulp was then washed thoroughly with tap water at pH 6 – 7 with the aid of a screened-bottom box. The freeness of the pulp was measured and when the value was greater than 230 ml, the pulp was subjected to refining. Sample pulp was then taken for fibre fractionation and fibre measurement. The rest of the pulp was formed into handsheets. Representative handsheets were then tested while all the others were subjected to further recycling. The cycle was continued until the fifth stage when there was only about 20% of the raw material left.

The material TMPN is a machine-made paper whose properties differ from handsheets. To be consistent with the make of paper, a separate portion of the original TMPN was, therefore, disintegrated and handsheets (TMPN #0) were prepared from it for use as reference material.

Measurement of Fibre Dimension and Pulp Freeness

Fibre length distribution was measured by the fractionation method using Bauer McNett Classifier (TAPPI Standard T234) (TAPPI). The fractionation method measures the average fibre length and length distribution directly by weight, with the aid of a fibre fractionator, which is a series of successive screens of decreasing mesh size (Mesh 14, 24, 48 and 100). The fraction for Pass Mesh 100 was calculated as the difference of these fractions from 100.

Fibre length was measured using a microscope (TAPPI Standard T232) (TAPPI). The microscope method consists of examining a slide of fibre under a microscope and measuring the length of every fibre.

Pulp freeness, which indicates the ease with which water drains away from the papermaking fibre, was determined following the TAPPI Standard T227 (TAPPI).

Determination of Handsheet Properties

Using the standard laboratory sheet former, handsheets with a basic weight of 43.5 g/m^2 were formed from the deinked pulp following that of the original material (TMPN). These were conditioned in the standard paper conditioning and testing room for at least four hours. Tests for burst, tear, and tensile strength properties were conducted according to the TAPPI Standards (TAPPI) T403 ts-63, T414 ts-64, T220 os-71, respectively.

Blending of Recycled TMPN with Virgin TMP, TMPN, or Unsorted Mixed Office Waste Paper

Using the proportions listed below, recycled pulp (TMPN #5) was mixed with TMP, TMPN #0, or UMOW; then handsheets were formed and tested:

Blends		Proportions
TMPN #5: TMP	=	90:10, 80:20, 70:30
TMPN #5: TMPN	=	90:10, 80:20, 70:30
TMPN #5: UMOW	=	90:10, 80:20, 70:30

Statistical Analysis of Data

The effects of number of recyclings and blending of fibre on the properties of the fibres and handsheets were evaluated statistically using

analysis of variance (ANOVA) in completely randomized design (CRD) and Duncan Multiple Range Test (DMRT).

RESULTS AND DISCUSSION

Properties of Virgin TMPN and Its Handsheets

It was expected that the machine-made TMPN would differ in properties from the handsheets made from re-slushing it due to differences in orientation of the fibre and several other factors. Table 1 shows these differences. The burst index readily lost about 40% of its original value (1.47 kPa.m^2/g). The lower burst index value of TMPN #0 might be due to the hornification of the fibre resulting from drying of the TMPN. This meant that the penetrability of hemicellulose to water had decreased. In this condition, the fibre did not have good contact with each other during consolidation of the sheet.

The higher values of tear and tensile indices, and folding endurance of TMPN # 0, the machine-made TMPN taken at the cross direction (CD), had to do with the orientation of fibre. The machine-made TMPN had short fibre concentrated in the CD and long fibre in the MD while the laboratory handsheet TMPN #0 consisted of uniformly distributed long and short fibre. Tear strength is dependent on the length of the fibre; the longer the bonded fibre, the harder is it for the paper to tear. There was a need for more force to be applied before the fibre broke due to the longer length. Similarly, the tensile strength and folding endurance of TMPN #0 might have benefited from the random formation of bonds between short fibre, and short fibre with long fibre.

The TMPN #0 values were used as reference for evaluating the effects and the critical level of recycling.

Effects of Recycling on Pulp/fibre Characteristics

Papermaking involves a series of unit operations which cause corresponding impact on fibre properties and consequently on paper properties. Thus, studies may be specifically on the impact of each operation or the impact of a collection of unit operations (Ellis & Sedlachek 1993).

This particular laboratory study attempted to produce paper from fibre after recyclings, done up to five times. Each recycling involved printing by mimeographing, re-pulping, deinking using a flotation cell followed by washing, refining, and handsheet-forming. The collective effect of the recycling process was evaluated based on the properties of the handsheets formed while the impact of each operation was traced to the fibre distribution and dimensions of the data.

Figure 2 shows the mesh fractions of TMPN before and after recycling. The longest fibre in Mesh #14 decreased continually with each recycling while the other fractions fluctuated. The greatest decrease (from 36.1 % to 14.99 %) or about 58%, was right after the first recycling. By the second cycle, the amount of fibre in Mesh #14 had been reduced by about 88 %. At the fifth cycle (TMPN #5), Mesh #14's long fibre

Table 1. Properties of TMPN and its handsheets.

Material	Freeness CSF (ml)	Basis weight (g/m^2)	Burst index (kPa.m^2/g)	Tear index (mN.m^2/g)	Tensile index (N.m/g)	Folding endurance
TMPN (MD)	193	43.48	1.47	4.04	48.07	38
(CD)				6.32	17.02	3
TMPN #0	193	42.46	0.88	8.13	27.64	5.09

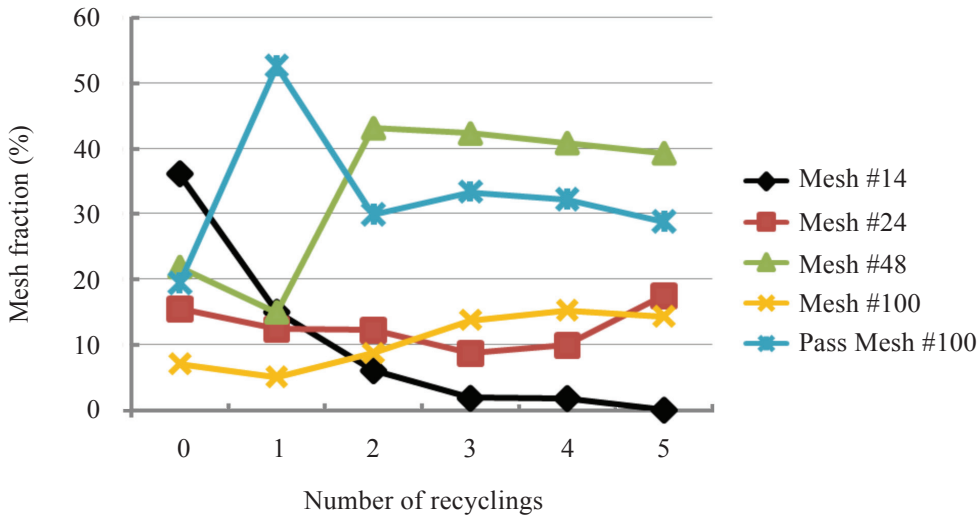

Figure 2. Percentage of each mesh fraction of TMPN fibre before and after recycling.

was virtually gone. The resulting increases in the other fractions due to breaking of the longer fibres were greatest with Mesh #48. Fibre passing the Mesh #100 screen were usually lost in the washing stage. Thus, starting from 1200 g of the original TMPN, the remaining material at the fifth stage was only about 200 g. Fibre fractionation data, therefore, gave an indication of the presence or absence of the long fibre in a certain material. This change in distribution of fibre properties was usually an impact due to screening, cleaning and flotation cells in the de-inking process (Ellis & Sedlachek 1993).

Table 2 shows the freeness of the pulp and the length of the fibre for each mesh fraction of TMPN before and after recycling. The pulp was refined to obtain freeness within a range not too far from the original material. In this case, the freeness range was targeted within 193ml – 230 ml. Thus, refining was not conducted in the first and fourth cycles.

Fibre length refers to the average length of the fibre and indicates how long or short the fibre is. These are important basis of information in evaluating the quality of the wastepaper intended for recycling.

Fibre were randomly selected and carefully noted as either of full length or of broken fibre. The amount of broken fibre ranged from 40 % to 80 %. Each material exhibited a decreasing trend in the length of fibre, whether full length or broken, with increasing mesh size. Each recycling generally caused a further decrease in fibre length of all mesh fractions. Modification of fibre dimensions were said to be effects of refining and dispersion (Ellis & Sedlachek 1993).

Effects of Recycling on Paper Properties

The effects of recycling on the properties of the handsheets are summarized in Table 3, which shows the treatment means for each property, after conducting tests for correlation with density, ANOVA and DMRT on the data.

Burst strength significantly decreased in the third cycle when much of the long fibre had been lost in the previous cycles. In the fourth cycle, burst strength decreased further as the fibre was not conditioned by refining. The burst index value recovered with refining in the fifth stage due possibly to fibrillation of fibre which may have increased the numbers of bonds of fibre and fibrils.

Table 2. Freeness and fibre length of TMPN before and after recycling.

Material	Freeness CSF (ml)		Mesh	Fibre length (mm)			
	Initial	Final		Full	Broken	Average	% Broken
TMPN #0	193	No refining	14	3.0610	2.361	2.7812	40
			28	2.5982	1.7885	2.1432	56
			48	2.2610	1.4686	1.8172	56
			100	1.1109	0.9000	0.9932	56
TMPN #1	217	No refining	14	2.9710	2.1364	2.6040	44
			28	2.6325	1.8640	2.2328	52
			48	2.5236	1.3409	2.0032	44
			100	1.8570	1.2747	1.5076	60
TMPN #2	410	Refined to 202	14	2.8130	2.1760	2.5580	40
			28	2.4936	1.8486	2.1324	56
			48	2.0478	1.1864	1.6688	44
			100[a]	–	–	–	–
TMPN #3	468	Refined to 200	14	2.240	1.8700	2.0624	48
			28	1.8431	1.4608	1.6596	56
			48	1.7814	1.1561	1.3312	72
			100	1.1820	0.6840	0.7836	80
TMPN #4	230	No refining	14	2.4500	1.5400	1.9412	56
			28	2.2418	1.4829	1.8168	56
			48	2.1000	1.0227	1.4536	60
			100	1.3390	0.8080	1.0204	60
TMPN #5	259	Refined to 211	14	2.2400	1.3880	1.8992	40
			28	1.4527	1.1586	1.2880	56
			48	1.2264	1.0628	1.1348	56
			100	0.7680	0.6340	0.6876	60

[a] Sample was inadvertently lost in handling.

Table 3. DMRT on the treatment means for each property of handsheets from TMPN and recycled TMPN.

Treatment (Number of recycling)	Freeness CSF (ml)	Burst index ($kPa.m^2/g$)	Tear index ($mN.\ m^2/g$)	Tensile index (N.m/g)	Folding endurance
TMPN 0	193	0.876 AB	8.128 B	27.643 A	5.090 A
TMPN 1	217	1.080 A	10.282 A	22.149 BC	6.163 A
TMPN 2	202	1.018 A	5.740 C	21.433 BC	1.623 B
TMPN 3	200	0.754 B	7.344 B	22.555 ABC	1.342 B
TMPN 4	230	0.682 B	10.192 A	20.085 C	1.378 B
TMPN 5	211	0.912 AB	7.500 B	25.474 AB	1.055 B

Means (in each column) followed by the same letter are not significantly different from each other.

Tear strength values at the first and fourth recyclings, where refining was not conducted, were higher than where refining was done and were even higher than in the original material. It is generally recognized that fibre length directly affects tear strength. Cross-reference with fibre length data in Table 2, however, showed a consistent decrease in values with every recycling. Thus, the high tear strength values at the first and fourth stages might have been due to factors other than fibre length. The original material used in this experiment was made from thermomechanical pulp (TMP). It has been reported that papers from TMP do not really show any significant change in tear strength with recycling (Chatterjee *et al*. 1993).

Tensile strength (measured as breaking length) has been reported to decrease the most after the first and second recycling, then on gradually in the succeeding recyclings (Ellis & Sedlachek 1993). In this experiment, the greatest decrease in tensile strength was after the first cycle. Succeeding cycles no longer caused a significant decrease in values compared with the first, indicating the positive effect of refining. Increase in bonding was said to be the main reason for the rise in paper tensile strength due to pulp beating or refining (Dasgupta 1994). The leap back in the fifth stage to almost the original value was rather unexpected. At this stage, there was almost no more long (Mesh 14) fibre (Figure 1 and Table 2). The compaction of the remaining shorter (Mesh 28 to 100) fibre probably resulted in an increase in the number of bonds and thus this effect was achieved.

In the case of folding endurance, there was no significant decrease in value after the first stage. The drastic decrease manifested from the second recycling indicated the brittleness of the paper and the washing out from the paper of chemicals that had been used for the original TMPN.

Based on the above data, the critical level of recycling might be at the fifth stage except for folding endurance. However, there would be a need also for a greater input of virgin or better quality fibre because refining also increases the volume of fibre which ultimately goes down the drain during the washing stage.

Effects of Blending Recycled Fibre with Virgin Fibre or Mixed Office Wastes

Recycled paper is actually the product of a mixture of different qualities of fibre. It may be composed of virgin pulp and recycled fibre from first, second, and or other stages.

This particular experiment was aimed to evaluate the properties of handsheets from mixtures of the preceding experiment's fifth stage recycled fibre (TMPN #5) with virgin pulp (TMP), with the original material TMPN, or with unsorted mixed office waste (UMOW). The material were disintegrated and simply mixed together. Table 4 summarizes the data obtained for this experiment after conducting ANOVA and DMRT.

The pulp blends showed no necessity for refining as the freeness values were within the desired range of 193 ml – 230 ml.

For burst index, each value was significantly different from the others. Although the highest value was obtained from the pulp blends with the greatest amount of virgin fibre (70 TMPN #5: 30 TMP), blending with UMOW generally performed better than blending with TMP or TMPN #0. Inconsistency in trend were observed with blends having 30% of either TMP or UMOW.

Tear and tensile indices were not significantly affected by pulp blend. This meant that the values did not differ significantly from each other. Compared with 100 TMPN #0, the tear index value was much higher than that of any of the treatment means. This indicated that blending recycled fibre with up to 30% of virgin fibre or UMOW could not compensate

Table 4. DMRT on the treatment means for each property of handsheets from recycled TMPN blended with unrecycled TMPN, UMOW, or TMP.

Treatment (Pulp blends)	Freeness CSF (ml)	Burst index (kPa.m^2/g)	Tear index (mN. m2/g)	Tensile index (N.m/g)	Folding endurance
TMPN 0	193	0.88	8.128	30.086	4.566
TMPN#5:TMPN#0					
90 : 10	202	0.65 G	4.02	25.78	1.49 C
80 : 20	193	0.77 EF	4.39	26.56	2.38 BC
70 : 30	211	0.72 FG	5.00	27.31	2.13 C
TMPN#5:UMOW					
90 : 10	220	1.12 BC	4.45	31.21	1.96 C
80 : 20	211	1.17 AB	4.33 A	28.12 A	3.77 A
70 : 30	220	0.74 FG	3.95	27.77	2.45 BC
TMPN#5: TMP					
90 : 10	193	0.89 DE	4.16	25.63	2.04 C
80 : 20	208	0.99 CD	4.42	28.32	2.36 BC
70 : 30	189	1.28 A	4.40	29.03	3.18 AB
TMPN# 5	211	0.912	7.50	25.474	1.055

Means (in each column) followed by the same letter are not significantly different from each other.

for the deleterious effect of recycling. Although the original value of the tensile index was higher than most of the treatment means, the difference was not significant. This justified the practice of blending recycled fibre with virgin or other better quality fibre. Folding endurance values likewise showed the beneficial effect of blending recycled fibre with other fibre. Much improvement was observed with a 20% to 30% % proportion of either TMP or UMOW. Even with only 10% of TMP or UMOW, the folding endurance had almost doubled.

CONCLUSION AND RECOMMENDATIONS

- A remarkable modification in fibre properties, from the original TMPN in every cycle of the recycling process, was observed. While refining was known to shorten the fibre, modification in distribution of fibre properties was attributed to the de-inking process.
- Recycling with refining generally shortens the fibre. Thus, the long fibre fraction decreases continually with each recycling while the other fractions increase. In this study, the greatest decrease, about 58%, came right after the first cycle. By the second cycle, the long fibre fraction was only about 12%.
- Refining generally improves the strength properties of paper from recycled fibre. With this recycling scheme, the critical level of recycling lasted up to the fifth stage but with barely 20% of the original material remaining.
- A proportion of about 10% to 30% of either TMP or UMOW was sufficient for the blending with recycled fibre to obtain strength properties within acceptable limits.

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