



**International Journal on Coconut R & D - Vol. 35 No. 1, 2019**

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<b>ICC Member Countries</b>	<b>US\$ 40.00</b>
<b>Non-ICC Member Countries</b>	<b>US\$ 50.00</b>

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## **Evaluation of Coconut Based *Anacardium occidentale* Agroforestry System to Improve the Soil Properties of Coconut Growing Lands in Wet, Intermediate and Dry Zone of Sri Lanka**

S. H. S. Senarathne and S. S. Udumann

### **Abstract**

This study was intended to assess the impact of coconut based *Anacardium occidentale* (Cashew) agroforestry systems on soil fertility of degraded coconut lands in wet, intermediate and dry zones of Sri Lanka. Two treatments were evaluated according to randomized complete block design with three replicates. Coconut based agroforestry systems intercropped with *A. occidentale* and sole coconut were evaluated as two treatments. Soils from three depths were analyzed for its' chemical, physical and biological properties.

According to the results, higher total N, available P and exchangeable K levels were shown in sole coconut systems than *A. occidentale* intercropped system while the higher total N levels (2% higher than top soil and 27% higher than deeper soil) were observed in sub soils compared top and deep soils. Higher P content was observed in top soils than in deeper soils. The exchangeable K was observed in higher quantities in sub soil than in deeper soils and was varied with locations. Organic matter content in intercropping of *A. occidentale* has been increased by 37% and the highest was observed in top soils. Soil bulk density has been reduced by 9% in *A. occidentale* intercropped system enhancing the root growth. Bulk density has been increased with the depth of the soil. Higher soil microbial activity was observed in *A. occidentale* intercropped system and it was 22% higher than sole coconut system. Sole coconut system has 50% higher soil moisture percentage and the highest was recorded in sub soils. This study confirms that intercropping of *A. occidentale* has a positive effect on improving soil fertility of degraded coconut growing soils in wet, intermediate and dry zones of Sri Lanka.

**Key words:** Agroforestry, Coconut, *A. occidentale*, Dry zone, Intermediate zone

### **Introduction**

Agroforestry is a form of land use that has long been practiced in many parts of the world (Regmi and Garforth, 2010) with the type and composition of tree species and their distribution and extent varying according to topography, biophysical attributes and the socio-economic conditions of the resource managers. Agroforestry is commonly understood as the integration of trees or deliberate retention of trees on agricultural land (Nair 1985) where the primary objectives are to produce food, fodder, fuel-wood and/or timber. There can also be co-benefits such as carbon sequestration, enhancing water quality, protecting soil and conserving biodiversity (Tamale *et al.*, 1995; Arnold 1997; Long and

Nair, 1999; Jose 2009; Alavalapati *et al.*, 2004). Sustainability of this farming system is reflected by its appropriateness in the given economic and environmental circumstances. Coconut is one of the most widely grown tree crops in the tropics, occupying in some regions up to 20-30% of the total cultivated area. With its economic life span of 60 years or more, it occupies the land for a long time. As a monocrop, its economic life in some places averages only about 40 years, depending on growing conditions (Burgess, 1981 and Opio, 1990). Beyond this period, the productivity of the land under coconut diminishes, and therefore, it is necessary to diversify the land use or to replant coconut.

The morphological characteristics of the coconut palm and the conventionally adopted spacing (ranging from 8x8 to 8.6x8.6m) associated with the coconut root system which normally clusters within 2 m of the stem allow open spaces for further cultivation and/or grazing. At the much used spacing of 8x8m, at least 64 m<sup>2</sup> is actually allocated to each palm and yet the effective root area per palm is only 12.5m<sup>2</sup> or 15.4% of the available space leaving 68.5m<sup>2</sup> (84.6%) of spacing underutilized. Furthermore, as established by Nelliath *et al.*, 1974, the top 30 cm of soil is generally devoid of functional roots and 86% of the coconut roots are found between 30-130 cm depth. This suggests that coconut is by nature suited to intercropping. The unique leaf canopy permits a large part of the solar energy to be transmitted through it. The percentage of light transmitted depends on the age of the palms, ranging from 20% under 10-20-year-old palms, to 50% in plantations of 40 years and over. In older plantations light transmission increases substantially, providing ideal conditions for coconut base farming systems (CBFS). To this extent, over 60% of smallholders practice CBFS. Intercropping is a major cropping system for coconut cultivation worldwide (Liyanage *et al.*, 1985; Magat, 2004; Ohler, 2007). Intercropping represents a more efficient use of natural resources and labour (Fordham, 1983); broadens farmer's income/ food security base and helps in weed control (Bonneau and Sugariato, 1999). There are many common annuals and

perennials recommended for CBFS. Using productivity as a land value measure suggests that coconut land generates a very low return per unit area compared to other crop land. But in the plantation sector, where long-term crops such as bananas, coffee, cocoa, pineapple, cashew, mango, etc. are involved, continuous cropping is commonly practiced (Fernando *et al.*, 1984).

Low land productivity in coconut plantations is highly associated with loss of fertile topsoil through accelerated erosion due to poor land management. Numerous studies have been undertaken to achieve this task through several agronomic practices, especially by improving fertility status of soil (Liyanage and Dasanayake, 1993). Incorporation of tree species producing substantial amounts of biomass is recognized as a solution for enhancing soil organic matter in a cost effective way and with alternative uses (Costa and Sangakkara, 2006).

However, for coconut, inclusion of a Cashew (*Anacardium occidentale L*) based agroforestry system is possible using available spacing efficiently. In addition to the more organic matter incorporation and mining nutrients from subsoil with its deep root system. Systematic incorporation of Cashew is an effective barrier for reducing the momentum of raindrops and overland flow but diminishing the risk of erosion. Cashew being a hardy crop can thrive well in variety of soils and is usually grown in poor soils where no other horticultural crop can be grown successfully. The major area under cashew cultivation is located in marginal waste lands, coastal laterite and sandy soils and as an intercrop with mature coconut plantations of Sri Lanka.

Cashew (*Anacardium occidentale L*), is one of the cashcrops which is grown with coconut in selected areas in Sri Lanka. Cashew is becoming an important cash crop for farmers in Sri Lanka where there is greater potential for increased production for the local market and export market. Presently, approximately 42,000 ha are under cashew plantations and around 10,000 MT of rawnuts are being produced which is only about 50% of the local demand (Weerakoon, 2011). Amongst the main areas of



cashew cultivation namely *Puttalam*, *Kurunegala*, *Batticaloa*, *Anuradhapura*, *Mannar* and *Hambanthota*, coconut cashew intercropping systems can be mainly identified in *Puttalam* district in commercial scale. Therefore, this study was design to assess the potential of using coconut based *A. occidentale* agroforestry systems to improve soil fertility of degraded coconut lands in wet zone, intermediate zone and dry zone of Sri Lanka.

### Materials and Methods

The study was conducted at Agronomy Division of Coconut Research Institute (CRI), Lunuwila, Sri Lanka, situated in North Western Province of Sri Lanka, (7° 20' 37" N, 79° 51' 42" E). Study was carried out in established experiment fields for intercropped coconut. The first field experiment was established at Rathmalagara Estate, Madampe in the low country intermediate zone (08° 02' N, 79° E; 35 m from mean sea level). Agro ecological zone of this area is IL<sub>1</sub> (Punyawardena *et al.*, 2003). Soils of this area belong to the Andigama series which categorized into great soil group of Red Yellow Podzolic (Mapa *et al.*, 2005) (Ferric Acrisols; FAO/ UNESCO, 1998). The mean annual rainfall and ambient temperature range were 1660 mm and 23.8°C - 30.4°C, respectively.

The second field experiment was established at Pallama Estate, Pallama in the low country dry zone. Agro ecological zone of this area is DL<sub>3</sub> (Punyawardena *et al.*, 2003). The experiment site represented the soils belongs to the great soil group of Red Yellow Podzolic (Mapa *et al.*, 2005) with soft or hard laterite (70-90%). The mean annual rainfall and ambient temperature range were 1200 mm and 28°C - 32°C, respectively.

Third field experiment was conducted at Walpita Estate, in low country wet zone. The soil at the site is Red Yellow Podzolic (RYP) (USDA soil taxonomy - Typic Rhodudults) (FAO/UNESCO soil taxonomy - Ferric Alisols) soils with soft and hard laterites (Mapa *et al.*, 2005). The area is characterized by bimodal pattern of rainfall with an annual mean precipitation of >1700mm, high ambient air and

soil temperature (24°C<sup>0</sup> - 29°C<sup>0</sup>) and bright sunshine hours (about 6-8 hours day<sup>-1</sup>). Reaction of the soil is slightly acidic (pH 4.0 - 4.5) throughout the soil profile. (Mapa *et al.*, 2005). In all locations, *A. occidentale* trees were cultivated in between coconut rows. Soils of coconut based *A. occidentale* agroforestry systems were evaluated through a soil fertility analysis by measuring soil physical, chemical and biological properties. Experiment was designed in a Randomized Complete Block Design (RCBD) with three replicates.

- T<sub>1</sub>. Coconut and Cashew (*A. occidentale*) mix cropping system
- T<sub>2</sub>. Sole coconut system (coconut was established with 8 m x 8 m spacing)

### Soil Sampling, preparation & analysis

In December 2013, three soil samples were randomly collected from 2.5m away from the effective coconut palms in each experimental plot at 0-15cm, 15-30cm and 30-45cm depths, respectively. Simultaneously, an undisturbed soil samples were collected using a core-sampler from desired depths (0cm, 15cm and 30cm) for bulk density determination. Samples were air dried separately at room temperature for 48-72 hours without any contaminations. Air dried soil samples were crushed and sieved through 2 mm sieve. Undisturbed soil samples were collected from same locations to determine microbial activity. For physic-chemical characterization the following soil parameters were considered: organic carbon of the samples were measured by Walkley-Black method (Walkley and Black, 1934); N was estimated by the Kjeldahl method (Jackson, 1973) and the P and K contents of the samples were analyzed by calorimetric method (Anderson and Ingram, 1993) and flame photometric method (Simard, 1993), respectively. As a soil biological property, microbial activity was determined by trapping CO<sub>2</sub> with alkali solutions, followed by the precipitation of carbonates with barium chloride and the titration of any remaining hydroxide with standardised acid (Stotzky, 1965).

### **Soil moisture content**

Soil samples were collected from four random points from the 2.5m away from the effective coconut palms a depth 0.3m, to determine potential treatment effects on soil moisture content during dry period (January or July). Collected samples were oven dried at 105°C to a constant weight and gravimetric soil moisture content was determined following IAEA (2008).

### **Data analysis**

Experimental data were analysed following Analysis of Variance (ANOVA) procedure using the statistical software SAS (SAS reference) and the significance of the differences between means was tested using Least Significant Differences (LSD) at  $P=0.05$  (SAS Institute 1999).

## **Results and Discussion**

### **Effect of cashew intercropping with coconut on soil physical properties (soil moisture and bulk density)**

The soil moisture content was determined during the dry periods in all three locations in February and March months in 2013. The soil moisture content of these three locations are shown in Table 1. The results showed considerable variation in soil moisture at different depths among three different locations. Sole coconut system had comparatively higher soil moisture content than coconut cashew intercropping system in all three locations. This was attributed to fact that cashew has a well developed deep tap root system which helps to absorb more water through deeper soil layers than coconut. According to the experiment results, the cashew coconut intercropping system had significant effect on soil moisture content at 15-30cm and 30-45cm soil depths but not at 0-15cm depth at Ratmalagara (intermediate zone) and Walpita (wet zone) locations. However, no significant effect on soil moisture content at all soil depths that were observed at Pallama (dry zone) location and it is mainly due to the loamy sandy textured nature of the soil.

The cashew intercropping system had a significant effect on soil bulk density at 0-15cm and 15cm-30cm soil depths, but not at 30-45cm soil depths at Ratmalagara and Walpita locations. However, no significant effect on soil bulk density at all selected soil depths at Pallama location where deep loamy sand textured soil can be found.

Comparatively lower soil bulk density was observed in cashew coconut intercropping system than sole coconut system (Table 2). This is mainly due to the fact that intercropping system contributes more organic matter content to the soil than sole coconut system. A high level of organic matter in the soil results in reduced bulk density, improved soil structure, aeration and highwater holding capacity all of which are attributes of a productive soil (Hsieh and Hsieh, 1990). Soil organic matter is responsible to a great extent, directly or indirectly for making the good physical environment in the soil and making it suitable for the growth of plant roots (Jeyamala and Soman, 1999). Application of organic matter has reduced the bulk density of the soil (Table 2), which is a vital soil characteristic for successful root development (Kuchenbuch and Ingram, 2004).

### **Effect of cashew intercropping with coconut on soil biological properties (soil microbial activity)**

The cashew intercropping system had a significant effect on soil microbial activity at 0-15cm soil depth at Ratmalagara and at 0-15cm and 15cm-30cm soil depths at Walpita (Table 3). However, no significant effect on soil microbial activity at all soil depths at Pallama. Higher soil microbial activity was observed in cashew coconut intercropping system compared to the sole coconut system and this is mainly due to the reason that intercropping practice contributes more organic matter to the system.

The application of organic matter to the soil is considered as a good management practice as it stimulates soil microbial growth and activity with subsequent mineralization of plant nutrients (Eriksen, 2005); and thereby, increase soil fertility and quality (Doran *et al.*, 1988).

**Table 1. Effect of growing cashew (*A. occidentale*) on soil physical properties (soil moisture)**

Treatments	Soil moisture (%)								
	Ratmalagara (intermediate zone)			Pallama (dry zone)			Walpita (wet zone)		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	10.88	8.46	6.23	9.23	8.11	7.18	12.54	13.44	8.95
T <sub>2</sub>	11.41	12.58	10.46	9.45	12.34	10.23	18.96	21.52	20.69
Significance	NS	*	*	NS	NS	NS	*	*	*
LSD (P<0.05)	-	3.34	3.81	-	-	-	5.42	6.78	9.12

\* Significantly different at P=0.05; NS- not significant

**Table 2. Effect of growing cashew (*A. occidentale*) on soil physical properties (soil bulk density)**

Treatments	Bulk density (g cm <sup>-3</sup> )								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	1.43	1.51	1.58	1.64	1.58	1.54	1.26	1.31	1.36
T <sub>2</sub>	1.71	1.78	1.64	1.76	1.65	1.72	1.45	1.42	1.41
Significance	*	*	NS	NS	NS	NS	*	*	NS
LSD (P<0.05)	0.18	0.22	-	-	-	-	0.12	0.08	-

\* Significantly different at P=0.05; NS- not significant

**Table 3. Effect of growing cashew (*A. occidentale*) on soil biological properties (soil microbial activity)**

Treatments	Microbial activity (mg/day)								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	69.4	55.7	56.8	34.4	26.7	18.4	88.2	94.5	66.7
T <sub>2</sub>	51.6	48.1	48.2	29.4	22.9	25.1	72.4	63.1	58.3
Significance	*	NS	NS	NS	NS	NS	*	*	NS
LSD (P<0.05)	10.2	-	-	-	-	-	8.6	23.1	-

\* Significantly different at P=0.05; NS- not significant

### **Effect of cashew intercropping with coconut on soil chemical properties (soil organic matter content, available P, total N and exchangeable K)**

#### **Soil organic matter content (%)**

The cashew intercropping system had a significant effect on soil organic matter content in all the locations. The highest values were observed at 0-15cm depth at Ratmalagara and Walpita locations (Table 4). Soil organic matter content of deeper soil layers showed different dynamic compared to top soil layers in three locations. Interestingly, soil organic matter content did not show any significant differences in sub soil from 15-30cm depth and 30-45cm depth in Pallama location.

This result reconfirms that organic inputs from cashew intercropping system have a constructive effect on soil organic matter content in soils of three locations. Moreover, Utomo *et al.*, (1990) and Reddy *et al.*, (2003) reported that SOM amelioration following leaf manure incorporation up to 1% of total soil mass. Nonetheless, substantial soil organic matter content were recorded from Rathmalagara and Walpita compared to the Pallama. This may associate to inherent low soil organic matter content in dry zone soils and rapid oxidation process in dry regions (Srinivasarao *et al.*, 2008). Cashew coconut intercropping system has showed more accumulation of organic matter in surface soil compared to subsoil in both locations lining with the study by Rudrappa (2006), who reported that soil organic matter was found stratified along the soil depth.

#### **Soil total nitrogen, available P and exchangeable K**

Mean values of soil nutrients at different soil depths under coconut cashew mixcropping systems in three different locations are presented in Tables 5, 6 and 7. The cashew intercropping system had no significant effect on soil total N at Ratmalagara and Pallama locations. However, significant effect has been shown on soil total nitrogen at top soil layers (0-15cm and 15-30cm) at Walpita location. In this location, higher total N content was observed in sole coconut planting

system compared to the cashew coconut intercropping system. The higher value in sole coconut system can be explained in terms of symbiotic relationship of the dense herbaceous undergrowth that releases or fixed nitrogen and rapid humification. The relatively lower mean values in mixed cropping may be attributed by inadequate application of nitrogen based chemical fertilizers, increasing immobilization by plants and leaching and volatilization which is common to most mineral soils (Jones and Weld, 1975; Brady and Weil, 2002).

Cashew growing with coconut had no significant impact on available phosphorus content in soil at three different locations (Table 6). However, available P content in soil was higher in sole coconut system and the value has been decreased with the soil depth. The higher content of available P in surface soil compared to sub soil can be ascribed to the accumulation of leaf litter besides supplementing the depleted nutrients through external sources. The lower phosphorus content in sub soil horizons might be attributed by the fixation of released P by clay minerals and oxides of iron and Aluminium (Leelavathi *et al.*, 2009).

The values of exchangeable K as revealed in Table 7, shows that the sole coconut system has comparatively higher K content compared to the coconut cashew mix cropping system. The exchangeable K content is generally lower for mixed cropping while the mono-cropping has a slight higher value. The variation in the base elements across the three sites will be reflected in the growth rate as well as the translocation and storage of carbohydrates and proteins into different plant parts. The comparatively lower values of this mix cropping system may be a reflection of losses through leaching, cultivation or harvesting (Jaiyeoba, 1995).

#### **Implications of the Study**

As a result of general reduction in the contents of the different soil properties under the two cropping systems (sole coconut and coconut cashew system), a significant variation in crop production is expected. The analysis of variance revealed that the mean of the some soil parameters were significantly different from

**Table 4. Effect of growing cashew (*A. occidentale*) on soil organic matter content (%)**

Treatments	Organic matter content (%)								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	2.45	1.59	0.95	0.84	0.78	0.47	2.66	2.35	1.04
T <sub>2</sub>	1.38	1.12	0.81	0.99	0.75	0.58	1.42	1.59	0.92
Significance	*	*	NS	*	NS	NS	*	*	NS
LSD (P<0.05)	1.02	0.27	-	0.14	-	-	0.98	0.37	-

\* Significantly different at P=0.05; NS- not significant

**Table 5. Effect of growing cashew on soil total N (ppm) content**

Treatments	Total nitrogen (ppm)								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	569.6	521.2	458.4	326.5	359.4	264.7	602.4	652.1	482.6
T <sub>2</sub>	572.4	541.3	486.1	374.6	402.5	285.1	634.8	721.2	504.4
Significance	NS	NS	NS	NS	NS	NS	*	*	NS
LSD (P<0.05)	-	-	-	-	-	-	29.5	17.26	-

\* Significantly different at P=0.05; NS- not significant

**Table 6. Effect of growing cashew on soil available P (ppm) content**

Treatments	Available P (ppm)								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	1.55	1.49	0.71	1.54	1.11	0.92	3.54	1.28	0.91
T <sub>2</sub>	1.73	1.52	0.84	1.62	0.98	0.86	4.18	1.41	1.11
Significance	NS	NS	NS	NS	NS	NS	NS	NS	NS
LSD (P<0.05)	-	-	-	-	-	-	-	-	-

\* Significantly different at P=0.05; NS- not significant

**Table 7. Effect of growing cashew on soil exchangeable K (meq/100g)**

Treatments	Exchangeable K content (meq 100g <sup>-1</sup> soil)								
	Ratmalagara			Pallama			Walpita		
	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm	0-15cm	15-30cm	30-45cm
T <sub>1</sub>	0.172	0.178	0.116	0.201	0.163	0.141	0.382	0.522	0.371
T <sub>2</sub>	0.181	0.191	0.137	0.269	0.181	0.157	0.431	0.596	0.402
Significance	NS	NS	NS	*	NS	NS	NS	NS	NS
LSD (P<0.05)	-	-	-	0.022	-	-	-	-	-

\* Significantly different at P=0.05; NS- not significant

each other in the three locations. This implies that the cropping systems constitute a threat to soil fertility and agricultural productivity. It also implies that different cropping systems effect changes in the content of the soil elements and the rate of nutrient immobilization. This will therefore require different management strategies to sustain soil fertility in this area.

### Conclusion

Generally, the study revealed that the contents of the soil chemical, physical and biological properties are vary with the cropping system and the location. However, cashew trees do not compete significantly with coconut palms for major soil nutrients (N, P and K) and this mix cropping system enhance the organic matter content of the soil. This system has significantly reduced the soil moisture content of the soil except in dry zone and as a result cashew and coconut yield can be affected negatively. Therefore, proper agronomic practices should be followed to conserve soil moisture as well as the soil fertility in coconut cashew intercrop mix cropping system.

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## Effect of Immersion in Calcium Chloride Solution on the Characteristic of Coconut Chips during Storage

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### Abstract

The quality of coconut chips can be increased, through efforts to improve processing by immersing the coconut meat in  $\text{CaCl}_2$  solution. The various concentrations of  $\text{CaCl}_2$  solution are 0.0%, 0.5%, 1.0%, 1.5%, and 2.0%. Furthermore, the effect of treatments was evaluated on the characteristic of coconut chips for 0 months, 2 months, 4 months, and 6 months of storage in plastic coated aluminum foil packaging. The results showed that coconut chips from DMT coconut meat with fruit 9 months old contain 2.36-2.49% moisture, 2.36-2.55% ash, 3.87-5.35% protein, 37.31-45.35% fat, 50.15-53.23% carbohydrate and 4.93-5.48% crude fiber. Immersion in  $\text{CaCl}_2$  solution and storage time increased the water content of coconut chips. The results of organoleptic testing showed that coconut chips still preferred by respondent up to 6 months of storage. The higher concentration of  $\text{CaCl}_2$  solution was used, resulting in smaller pressure (gram force) to break or destroy coconut chips, which can be interpreted that coconut chips have a crispness that is still good. Next, the color measurement uses Chromameter Konica Minolta CR-400, L (Lightness) value to 6 months ranged from 76.39-77.65, which indicated that the color of the product is still predominantly bright white.

**Key words:** Coconut chips, quality, storage

### Introduction

The rapid development of the population in Indonesia has forced the government to make various breakthroughs to provide food reserves especially rice (Rindengan, 2018). Rice can supply approximately 50% calorie needs from the recommended calorie standard of 2200 kcal/capita/day (Susenas, 2003 in Nainggolan, 2004). As a coconut producing country, coconut has actually the potential to be utilized in providing some of the daily nutritional needs.

In Indonesia, consuming snacks has become a separate lifestyle especially in urban communities. It is shown by the increasing number of types of snacks circulating in traditional markets and supermarkets. Hence, a portion of calorie requirement could be fulfilled by consuming snacks. Raw materials used for snacks processing, for example, are tubers, fruits, and livestock by-product like skin. Besides that, it can also come from coconuts, such as by -product of coconut oil processing with wet extraction, namely coconut pulp and 9 months aged young coconut meat (Rindengan *et al.*, 2004).

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Coconut is currently only considered as a source or raw material for making cooking oil, actually neglecting its potential as a source of alternative nutritional components (Rindengan, 2018). Other than using it as raw material for producing cooking oil and virgin coconut oil, coconut meat can also be a source of protein, carbohydrate, fiber, vitamins, and minerals. Coconut meat can be classified as a good source of protein because it does not contain antinutrient compounds such as those found in legumes. In addition, coconut meat from young and mature nuts, has a fairly good nutritional composition so that it can be utilized for processing a variety of appropriate food products. Coconut meat with fruit age 9 months of GKB x DMT hybrid contains 73.66% water, 1.81% ash, 2.50% protein, 13.06% fat, 8.97% carbohydrate, and 5.05% crude fiber (Rindengan, *et al.*, 1996). Besides that, it has a composition of omega 9 fatty acids or oleic acid (C18:1, n-9) 7.95% and omega 6 or linoleic acid (C18:2, n-6) 1.81% (Rindengan, 2002). Omega 9 fatty acids are very important for maturation of the functions of brain nerve cells, which mostly occur from birth to the fourth age. While omega 6 is an essential fatty acid that is very necessary from the time of conception until the first two years of the child's age (Anonim, 2002). According to FAO and WHO, the standard of additional food for infants and children must contain linoleic acid (omega 6) 1.4gr/100 gr.

In Balitpalma (IPCRI), the processing of snacks/ coconut chips use an oven dryer at 90°C for 2 hours and then continued at 70°C for 3 hours to reduce water content of coconut chips (Rindengan, *et al.* 2004), without the addition of Food Additives to improve the crunchy properties and storability of the product. Food additives like calcium chloride (CaCl<sub>2</sub>) as calcium salt is soluble in water and is extensively used to improve the texture of processed fruit and vegetable products. It is also utilized to obtain the crispy texture of chips because it can reduce the decomposition of cells which cause softening of the tissue. Calcium chloride can also inhibit the growth of microorganisms (Tuwitii, 2010).

The result of Sari's research (2010), showed that the highest quality of papaya chips is obtained with immersion in 1.5% CaCl<sub>2</sub>. On the other hand, according to Nisak (2007), the highest quality of papaya chips is obtain with immersion in CaCl<sub>2</sub> solution for 40 minutes. In the Philippines, coconut chips can be processed at age 9-10 months and 12 months. This product has been exported to various countries, including Germany, Sweden, Canada, and Denmark with price range \$115 US to \$1562 US (Masa and Montecillo, 2002). The objective of the research was to determine the influence of immersion in CaCl<sub>2</sub> on the characteristics of coconut chips during storage.

## Materials and Methods

### Materials

The materials used are young coconut meat (age 9 months) of Mapanget Tall (DMT) coconut variety. Mapanget Tall (DMT) coconut variety originated from Mapanget District, North Sulawesi Province, Indonesian. The description of Mapanget Tall (DMT) coconut is an age of first flowering 5 years, age of first harvesting 6 years, number of bunches 12-14/palm/year, number of nuts 90/palm/year and 12.870 nuts/ha (Balitpalma, 2018).

### Methods

#### *Processing of Coconut Chips*

The coconut meat is separated from the shell and then the testa layer was removed. Then it is thinly sliced to about 4-5 cm long using knife. The sliced coconut meat is immersed in CaCl<sub>2</sub> solution for 12 hours. The concentration of CaCl<sub>2</sub> solution are a1) 0.0%, a2) 0.5%, a3) 1.0%, a4) 1.5%, and a5) 2.0%. Then, drained and added with 20% sugar solution, boiled for 30 minutes and dried using oven Memmet UFB-500 type. Drying is done at temperature of 90°C for the first 2 hours and then reduced to temperature of 70°C for succeeding 3 hours (Rindengan, *et al.* 2004).

Furthermore, the coconut chips are packaged using aluminum foil (plastic coated) making it easier to glue. Then, packed coconut chips are stored for 0 months (b1), 2 months

(b2), 4 months (b3), and 6 months (b4). The research was conducted in two replications.

### Evaluation of Coconut Chips Quality

Moisture content, protein, fat, ash, and crude fiber were determined following the standard methods of the Association of Official Analytical Chemist (1995), starch (Anthrone Method, AOAC 197), carbohydrate content was determined by subtracting of 100% with the total sum of water content, ash, protein, and fat in sample (Winarno, 1986). Organoleptic test (color, flavor, taste, and crispness) with scale 1 = really dislike, 2 = dislike, 3 = normal, 4 = like, and 5 = really like, use 20 respondents (Soekarto, 1985). Color analyzed by Chromameter method (Ramsey, 2012) using Chromameter Konica Minolta CR-400 type and The crispness using Texture Analyzer (Peleg and Bagley, 1983).

### Statistical Analysis

Observations data were analyzed using SPSS 16.0. If there is a difference between treatments followed by DMRT Test (*Duncan Multiple Range Test*)

## Result and Discussion

### 1. Composition of Raw Material and Coconut Chips

The composition of DMT coconut meat from 9 months old fruit which was used for making coconut chips is shown in Table 1. The crude fiber content of 4,58% is a good component as a raw material for snacks. Hence, the product produced is expected to have high crude fiber which can help the metabolic process in the body.

The composition of coconut chips at immersion treatments in  $\text{CaCl}_2$  solution with concentrations of 0-2.0% are shown in Table 2. The composition of coconut chips immersed in  $\text{CaCl}_2$ (0-2.0%) solutions has moisture contents ranging from 2.36-2.49%, ash 2.36-2.59%, protein 3.87-4.65%, fat 37.31-40.25%, carbohydrate 50.13-53.23%, crude fiber 4.93-5.48%, while starch is not detected. According to Butler (2019), 6 grams of 10 grams carbohydrate contained in coconut flour is dietary fiber, while

the remaining 4 grams is crude fiber and soluble fiber. Therefore, starch is not detected as well as amylose.

Based on the results obtained, all the nutritional components of coconut chip were increased compared with the raw material (fresh coconut meat) because the water content in fresh coconut meat of about 70% has evaporated at the drying process, so that the nutrient component is concentrated.

The composition of coconut chips produced by Coconut Development Board, Kochi-India with coconut meat at age 8-10 months as raw material and the composition (per 20 gram) is fat 9.42 gram, lauric acid 4.82 gram, crude fiber 1.93 gram, calcium 2.10 gram, iron 1.30 mg, and cholesterol 0.00 gram (Anonim, 2019). While, the composition of coconut chips (per 100 gram) produced from Thailand is energy 77 calories, fat 3.6 gram, protein 1.4 gram, fiber 3 gram, carbohydrate 10.3 gram, calcium 43 mg, phosphorus 56 mg, iron 1.0 mg, and vitamin C 6 mg (Anonim, 2005). On the other hand, according to Sanchez, *et al.* (1996), the composition of dried buko chips is 4.7% moisture content, 3.6% protein, 17.7% fat, and 61.6% carbohydrate for sweetened type. The unsweetened type has a composition of 2.3% moisture content, 7.7% protein, 51.4% fat, and 13.6% carbohydrate. Differences in the results obtained may be caused by raw material used, processing, and use of food additives. In Indonesia, the standard quality for chips is based on the raw materials used, but the one for coconut chips is not yet available.

### 2. Moisture Content of Coconut Chips

Statistic analysis results show that the concentration of  $\text{CaCl}_2$  solution has effect on the level of coconut chips' water content (Figure 1). Moisture content at immersion in 0-1.5%  $\text{CaCl}_2$  solution is 1.47-1.76%, and increased to 2.52% at immersion in 2%  $\text{CaCl}_2$  solution. This is due to the hygroscopic characteristic of  $\text{CaCl}_2$  (Kemp, *et al.* 2000). Hence, the water content of coconut chips increases with the higher concentration of  $\text{CaCl}_2$  solution.

**Table 1. Composition of DMT coconut meat with fruit age of 9 months**

No.	Observed	Unit	DMT <sup>a)</sup>	DMT, fruit age 12 months <sup>b)</sup>
1.	Moisture content	%	72.90	49.80
2.	Ash	%	0.64	-
3.	Protein	%	2.37	3.32
4.	Fat	%	9.09	20.21
5.	Carbohydrate	%	15.00	5.37
6.	Crude fiber	%	4.58	2.81

Note: <sup>a)</sup>Fresh coconut meat without testa<sup>b)</sup>Tenda, *et.al* (1997)**Table 2. Composition of coconut chips immersed in CaCl<sub>2</sub> (0-2.0%) solution<sup>\*)</sup>**

No.	Observation	Unit	CaCl <sub>2</sub> (%)				
			0	0.5	1.0	1.5	2.0
1.	Moisture content	%	2.42	2.45	2.48	2.49	2.36
2.	Ash	%	2.59	2.55	2.47	2.39	2.36
3.	Protein	%	4.45	4.65	5.35	3.87	4.32
4.	Fat	%	37.31	37.55	39.57	39.29	40.25
5.	Carbohydrate	%	53.23	52.80	50.13	51.96	50.71
6.	Crude Fiber	%	5.30	5.48	4.93	5.32	5.12
7.	Starch	%	0	0	0	0	0

<sup>\*)</sup> after drying and cooling**Table 3. Effect of immersion in CaCl<sub>2</sub> solution on characteristics organoleptic test of coconut chips**

Immersion in CaCl <sub>2</sub> solution	Organoleptic Test			
	Color	Flavor	Taste	Crispness
0.0%	3.57a	3.63a	4.01a	3.95a
0.5%	3.69a	3.38a	3.89a	3.84a
1.0%	3.66a	3.41a	3.76a	3.97a
1.5%	3.69a	3.35a	3.73a	3.74a
2.0%	3.63a	3.45a	3.86a	3.82a

Note: Numbers followed by the different letter at the same column are significantly differenced at 5% of DMRT

According to Sanchez, *et al.* (1996), the moisture content of dried buko chips for sweetened type and unsweetened type is 4.7% and 2.3%, respectively. The Philippine standard for fancy cut desiccated coconut for which coconut chips can be categorized specifies a moisture content below 4 %. However, for the other types of desiccated coconut, the Philippine standard stipulates a maximum moisture content of 3% (PCA Administrative Order No. 003, Series of 1981). Based on Figure 1, Immersion in 2%  $\text{CaCl}_2$  solution increased the moisture content of coconut chips to 2.52% but it's still within the limit of the maximum set for desiccated coconut.

Figure 2 showed that the length of storage time has an influence on the moisture content of coconut chips. The moisture content of coconut chip up to 6 months of storage is 2.2%. Nevertheless, it is still far too compared than the standard of the moisture content of cassava chips (6.0%) and dried buko chips for sweetened type and unsweetened type is 4.7% and 2.3%, respectively (PCA Administrative Order No. 003, Series of 1981).

### **3. Characteristics of Coconut Chips**

#### **3a. Organoleptic test**

Value of organoleptic test is scale 1 = really dislike, 2 = dislike, 3 = normal, 4 = like, and 5 = really like. Effect of immersion in 0-2%  $\text{CaCl}_2$  solution and storage time on the results of color, flavor, taste, and crispness value of coconut chips can be seen in Table 3 and Table 4.

Based on Table 3, immersion in 0-2.0%  $\text{CaCl}_2$  solution has no effect on the organoleptic test of coconut chips. Values for colors range from 3.57 to 3.69 (normal to near like), flavor 3.35-3.63 (normal to near like), taste 3.76-4.01 (close to likes), and crispness 3.74-3.97 (close to likes). Furthermore, in Table 4, the storage time did not have an effect on flavor and taste of coconut chips. Range of flavor values 3.44-3.56 (normal to near like) and taste 3.75-3.91 (normal to near like). However, color and crispness of coconut chips are influenced by storage time. Color values of the organoleptic test at

storage time up to 6 months 3.80 (close to like) and crispness 4.09 (like). The crispness of coconut chips that lasts until 6 months of storage, probably caused by using packaging materials made of plastic coated aluminum foil (Figure 3), so the product is protected.

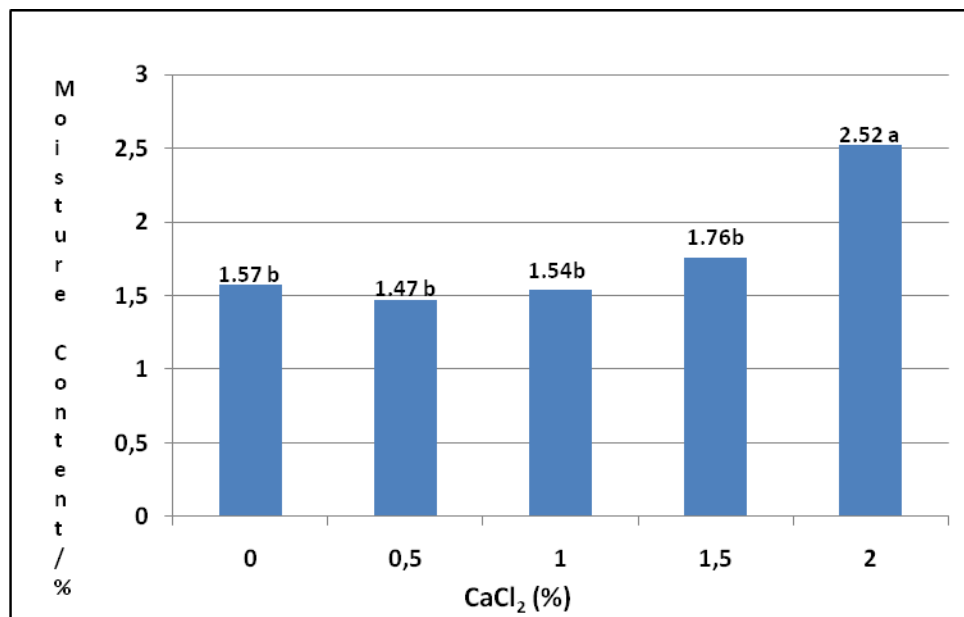
#### **3b. Crispness analysis using texture analyzer**

The crispness of coconut chips can be measured by the organoleptic test and using crispness measuring devices i.e. Texture Analyzer. The results of the texture analyzer are shown in Figure 4. Break values of coconut chips influenced by the concentration of  $\text{CaCl}_2$  but storage time not affected. Based on Figure 4, higher of  $\text{CaCl}_2$  concentration in immersion, the break values decreases. This proves that only a smaller pressure is needed in gram force for a break or destroy the coconut chips, which can be interpreted that coconut chips have a good crispness. Crispness values of organoleptic test not influenced by  $\text{CaCl}_2$  concentration but a range of crispness values 3.82-3.97% (normal to near like).

The measurement results crispness of cassava chips without treatment (control) were performed by Ginting (2014) is 500 gram force compared than the results obtained of immersion in 0-20%  $\text{CaCl}_2$  solution give a range break values only 100.39-137.73 gram force, that means coconut chips need only a smaller pressure in gram force compared than cassava chips. This might also because by a characteristic of raw materials used, coconut meat not contain starch, while cassava contains 73% starch.

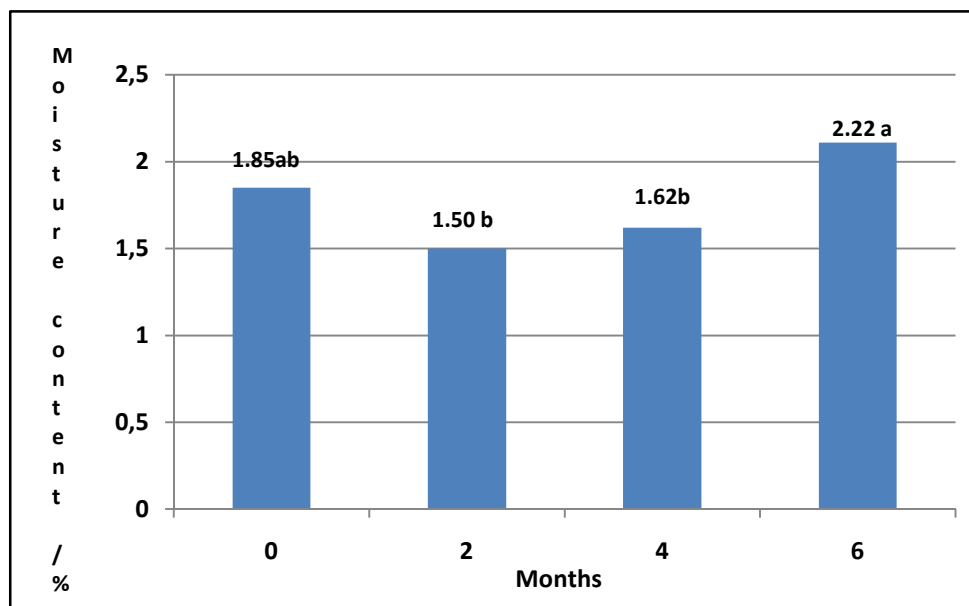
#### **3c. Measuring the color of coconut chips using Chromameter Konica Minolta CR-400**

The measurement of color using a Chromameter Konica Minolta CR-400 obtained values like L (Lightness: between 0-100 is white), a (range red and green color 0-60), and b (range yellow and blue color 0-60). The results of measuring the color of coconut chips showed in Table 5.



Note: Numbers followed by the different letter at the graph are significantly differenced at 5% of DMRT

**Figure 1. Effect of immersion in CaCl<sub>2</sub> solution on the moisture content of coconut chips**



Note: Numbers followed by the different letter at the graph are significantly differenced at 5% of DMRT

**Figure 2. Effect of storage on the moisture content of coconut chips**

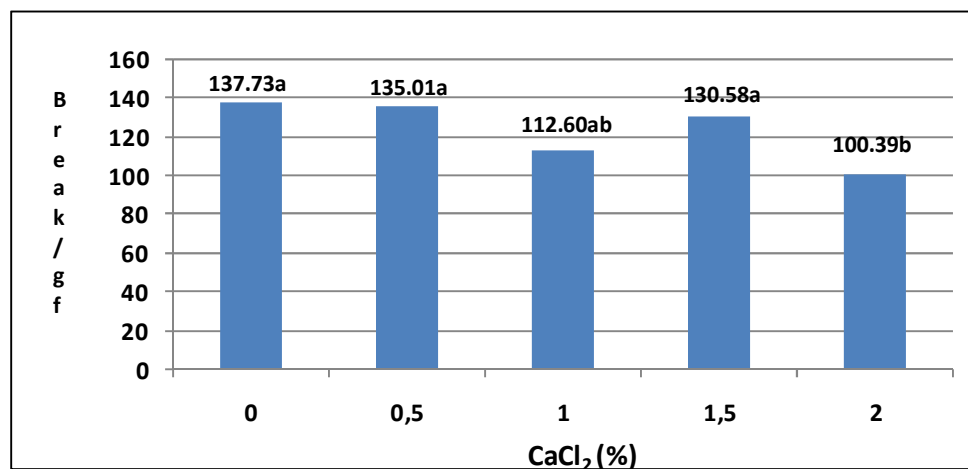
**Table 4. Effect of storage time on the characteristic of coconut chips**

Storage Times	Organoleptic Test			
	Color	Flavor	Taste	Crunchy
0 months	3.41b	3,56a	3.91a	4.06a
2 months	3.65a	3.44a	3.85a	3.73b
4 months	3.73a	3.33a	3.75a	3.58b
6 months	3.80a	3.44a	3.88a	4.09a

Note: Numbers followed by the different letter at the same column are significantly differenced at 5% of DMRT



**Figure 3. Coconut chips packed with plastic coated aluminum foil**



Note: Numbers followed by the different letter at the graph are significantly differenced at 5% of DMRT

**Figure 4. The break values of coconut chips using Texture Analyzer**

**Table 5. The color of coconut chips using Chromameter Konica Minolta CR-400**

Treatment		Color Characteristics		
Concentration of CaCl <sub>2</sub> (%)	Storage (months)	L	a	b
0.0	0 months	77.40	4.13	4.17
0.5		76.27	4.25	3.93
1.0		74.45	3.75	5.15
1.5		75.85	3.99	4.00
2.0		77.06	3.98	3.33
0.0	2 months	75.23	2.98	3.40
0.5		74.36	3.06	3.15
1.0		73.28	2.80	5.02
1.5		73.94	2.78	3.58
2.0		73.88	2.81	3.46
0.0	4 months	74.46	4.10	4.93
0.5		74.87	2.26	2.55
1.0		73.76	3.91	3.85
1.5		73.97	4.02	3.49
2.0		74.09	3.75	3.19
0.0	6 months	77.65	3.19	3.21
0.5		77.33	3.36	3.25
1.0		77.35	2.97	3.97
1.5		76.76	3.09	2.88
2.0		76.39	3.14	2.78

The results of coconut chips color measurement with immersion in (0-2%) CaCl<sub>2</sub> solution:

1. storage time for 0 months, L values 74.45-77.40; a (Red and green color between 3.75-4.25); and b (yellow and blue color between 3.33-5.15).
2. storage time for 2 months, L values 73.28-75.23; a (Red and green color between

2.78-3.06); and b (yellow and blue color between 3.15-5.02).

3. storage time for 4 months, L values 73.76-74.87; a (Red and green color between 2.26-4.10); and b (yellow and blue color between 2.55-4.93).
4. storage time for 6 months, L values 76.39-77.65; a (Red and green color between 2.97-3.36); and b (yellow and blue color between 2.88-3.25).



Based on an organoleptic test (Table 3), the color of coconut chips is 3.57-3.69 (normal to near like) not influenced by  $\text{CaCl}_2$  concentration but influenced by the length of storage with range values 3.41-3.80 (normal to near like). The results of measurement coconut chips color at immersion in (0-2%)  $\text{CaCl}_2$  concentration using Chromameter Konica Minolta CR-400 Showed that the product is still predominantly bright white (Figure 3) with a value up to 6 months between 76.39-33.65. While, a value between 2.97-3.36 and b 2.78-3.97. The a and b values still lower compared than range value for a (red and green color 0-60) and b values (yellow and blue color 0-60). This might because by temperature drying below  $100^\circ\text{C}$ , so the caramelization process is avoided. Ginting (2014) reported that the color of cassava chips without immersion in acetic acid solutions have an L value 62.06, a 1.61, and b 27.12. This is due to processing with the deep fat fryer system at  $170^\circ\text{C}$  for 1.45 minutes, so that the caramelization process occurs.

### Conclusion

The coconut chips obtained by processing coconut meat from 9 month old DMT coconut variety contain 2.36-2.49% moisture, 2.36-2.55% ash, 3.87-5.35% protein, 37.31-45.35% fat, 50.15-53.23% carbohydrate, and 4.93-5.48% crude fiber. Immersion in  $\text{CaCl}_2$  solution and storage time increased the water content of coconut chips, but still far from the maximum limit on commercial chips. Based on the organoleptic test, immersion in 0-2%  $\text{CaCl}_2$  solution does not have any effect on color, flavor, taste, and crispness of coconut chips. Likewise, the storage time did not affect the flavor and taste of coconut chips, but color and crispness changed during storage.

The crispness of coconut chips using Texture Analyzer influenced by immersion in  $\text{CaCl}_2$  solution. The higher concentration of  $\text{CaCl}_2$  solution used in immersion, the break value decreases. This proves that only a smaller pressure is needed in gram force for a break or destroy the coconut chips, which can be interpreted that coconut chips have a good crispness. The measurement of color using a Chromameter Konica Minolta CR-400 showed that the product is still predominantly bright white

because of white value up to 6 months between 76.39-33.65.

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## Coir Pith – A Medium for Oil Absorption

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### Abstract

Coir pith, the byproduct of coconut husk, due to its abundance nature and its porous structure can be effectively used for oil adsorption. Modification of coir pith to make as hydrophobic may allow them to be used for oil adsorption. Oil spills can destroy marine aquatic life and have a great impact on environment. In this study coir pith have been treated enzymatically (Lipase, Protease & Glucanase) and chemically (Acetylation) to impart hydrophobicity and to enhance oil adsorption capacity. The coir pith samples were characterized periodically by FTIR, SEM. The extent of acetylation was evaluated by weight percent gain. The results suggests that acetylated coir pith could be beneficial in oil adsorption and potentially provide a low cost environmentally friend adsorbent for oil spill.

**Key words:** Coir pith, Lipase, Protease, Glucanase, Acetylation, Oil adsorption capacity.

### Introduction

Lignocellulose biomass, comprising primarily of forestry, agricultural and agro-industrial wastes constitute an abundant, renewable inexpensive energy source (Kavitha and Namasivayam, 2007). Lignocelluloses are complex biomass, where carbohydrates such as cellulose and hemicelluloses are tightly bound to lignin molecules. The use of lignocelluloses as a sorbent in oil spillage is gaining acceptance in recent times (Bodirlau and ATeacaRom 2009, Moses *et al.* (2004). The major constituents of the lignocelluloses are cellulose, hemicellulose and lignin. Cellulose is  $\beta$ -(1-4) linked polymers of glucose made of cellobiose units with about 2000 to 27000 glucose residues. These chains are packed by hydrogen bonds in so called “elementary fibrils” originally considered to be 3-4 nm wide and contain about 36 chains. These elementary fibrils are then packed in so called micro fibrils. These fibrils are attached to each other by hemicelluloses, amorphous polymers of different sugars as well as other polymers such as pectin and covered by lignin. The micro fibrils are often associated in the form of bundles of macro fibrils (Taherzadeh and Karimi 2007, Delmer and Amor 1995). The high resistance to natural degradation by microorganisms is mainly due to its complex chemical structure. Sometimes, accumulation of slowly decaying lignocellulose wastes also causes severe environmental hazards.

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Coir pith is a lignocellulosic by-product produced during extraction of coir fiber from coconut husks and constitutes a readily available, abundant and renewable biomass. The coir pith has been experimented as a substrate for de-ionizing waste water, substrate for the production of bio fertilizers, biopolymers and artificial soil for plant growth (Namasivayam *et al* (2001). Recently, the coir pith has been investigated as a substrate for bioethanol production owing to their high cellulose content. Detailed laboratory studies have established its utility as an efficient oil absorber, with potential application in combating oil spills. The ZnCl<sub>2</sub> activated coir pith carbon was reported to be an efficient adsorbent for inorganic anions such as nitrate, thiocyanate, selenite, chromium (VI), vanadium (V), sulfate, molybdate, phosphate and heavy metals such as nickel (II) and mercury (II). Natural coir pith, comprising mostly of cellulose and hemi-cellulose is hydrophilic and an inefficient adsorber of lipophilic oil residues. Hence several chemical and biological approaches were envisaged to make coir pith more hydrophobic, thereby enhancing its oil adsorption capability. The present paper attempts at chemical and biological approaches for enhancing the oil adsorption properties of coir pith. The notion of the reactions are intended to disorganize the crystalline structure of macro and micro fibrils, in order to release the polymer chains of cellulose and hemicelluloses, and/or modify the pores in the material to enhance the oil adsorption properties.

Biological treatment involved primarily enzymatic modifications with suitable industrial enzymes like protease, lipase and glucanase. Chemical treatments included acetylation (Bledzki *et al* (2008), Diao She *et al* (2010), Mohd. Ghazalimohd nawawi *et al* (2008) acid hydrolysis, Ketal formation with MIBK, acetone, etc. It was speculated that suitable derivatization of the free hydroxyl groups (of cellulose and hemi-cellulose) would reduce the hydrophilicity of coir pith rendering it a good adsorbent for hydrophobic liquids like crude petroleum oil. In addition, coir pith can potentially provide a low cost environmentally friendly adsorbent for oil spill cleanup.

## Material and Methods

**Material collection:** Coir pith for the experimental was collected from the coir fibre extraction units in and around Alappuzha. The chemicals used for the study were of Sigma Aldrich grade.

### Effect of particle size on Oil adsorption properties of Coir pith:

For the present study the coir pith was fractionated based on their size into >0.5mm and 0.5 to 1mm size particles, freeze dried to constant weight and the oil adsorption efficiency was monitored. For measuring the oil adsorption properties, one gram dried coir pith was mixed with used-engine oil and the volume of oil adsorbed was calculated gravimetrically.

### Biological treatment: Enzyme treatment studies

**Protease treatment:** Proteases break up proteins into smaller peptide fragments. Proteases generally promote the hydrolysis of a peptide by activating a nucleophile, polarizing the peptide carbonyl and stabilizing the tetrahedral intermediate. Protease like all enzymes is very specific so recognizes side chain to know where to cleave. Coir pith samples were treated with protease enzyme for the time period of 1, 2, 3 & 4 hours and the enzyme treated samples were analyzed for the oil adsorption efficiency.

**Lipase treatment:** Lipase includes enzymes that hydrolyze lipids, fatty acids, and acyl glycerides, including phosphoglycerates, lipoproteins, diacylglycerols, and in plants, lipids are used as structural components to limit water loss and pathogen infection. These lipids include waxes derived from fatty acids, as well as cutin and suberin. Coir pith samples were treated with lipase enzyme for different time intervals (1, 2, 3 & 4 hours) and the oil adsorption efficiency of the treated samples was determined.

**Glucanase treatment:** Glucanase enzymes promote the hydrolysis of  $\beta$ - glycoside linkages in the cellulose. The coir pith sample was treated with glucanase enzyme for varying time intervals and the oil adsorption efficiency is evaluated gravimetrically.

**Chemical treatment 1. Acetylation:** Figure 1 shows process of Acetylation which was carried out by refluxing coir pith with acetic anhydride and pyridine (10:1) ratio for varying time durations (3, 6 12, 18 & 24 hours). Oil absorption efficiency of the samples was evaluated gravimetrically.

## 2. Estimation of acetate groups in acetylated coir pith

The percentage of acetyl groups in different samples was determined by saponification method. 0.1g of samples was saponified with aq. NaOH solution for thirty minutes. The filtrate analyzed for the excess NaOH remaining after saponification by titrimetric methods. The amount of sodium hydroxide consumed for saponification proportional to the amount of acetylated group in the samples.

**3. Acid hydrolysis:** The Acid hydrolysis was carried out by using 12% HCl for 48 hours.

## 4. Ketal derivative formation:

**a. With acetone:** Figure 2 shows the reaction of coir pith sample with acetone in presence of FeCl<sub>3</sub> catalyst refluxed for 24 hours.

**b. With MIBK:** The coir pith sample was refluxed with Methyl isobutyl ketone (MIBK) in presence of catalytic amount of the FeCl<sub>3</sub> for 48 hours.

## 5. Methanolysis

Figure 3 shows the process of Methanolysis of coir pith was carried out by refluxing coir pith with methanol and catalytic amounts of conc. HCl for 32 hours.

## 6. Preparation of Activated Carbon from coir pith

Activated carbon is produced by anaerobic heating of coir pith at 435°C in a muffle furnace for a time period of 2 hours. The physical properties like pH, particle density, bulk density and porosity of the activated carbon were determined using standard procedures (Kumar

and Chinnaiya 2009, Franklin e. Barton *et al* (1988). The Infra red spectrum of samples were recorded in **SHIMADZU FT-IR Instrument (Model: IR Prestige 21)** while the SEM analysis was carried out by **TESCAN VEGA3**. Oil adsorption studies of coir pith and modified coir pith samples were determined gravimetrically as per standard procedure.

## Calculation of oil adsorption efficiency

The oil adsorption efficiency of natural coir pith and treated samples were determined gravimetrically by **ASTM F 726** method. The efficiency of oil adsorption of different samples was calculated using the equation:

$$\text{Oil adsorption efficiency } (\%) = \frac{N_1 - N_0}{N_0} \times 100$$

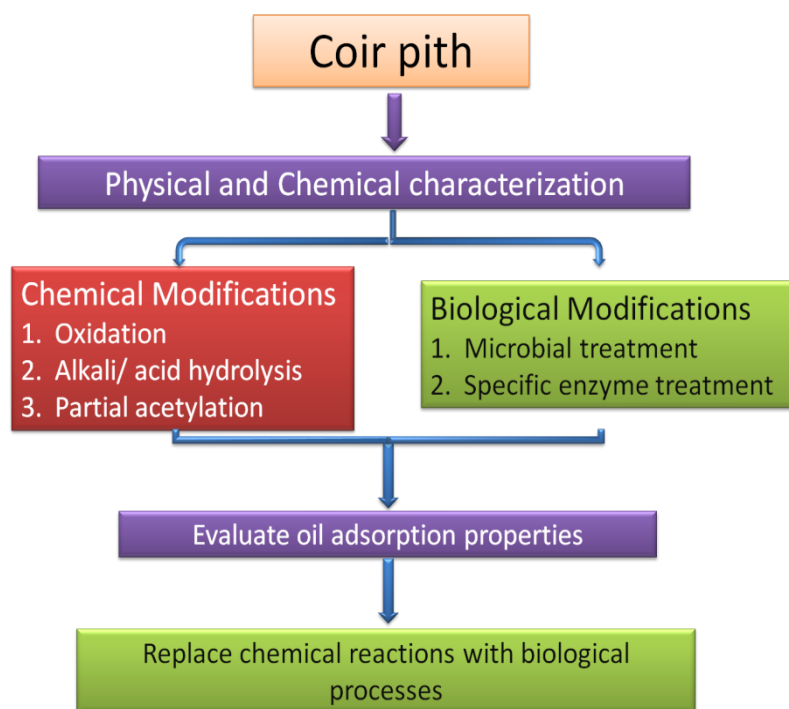
Where N<sub>1</sub>- weight of coir pith after oil adsorption

N<sub>0</sub>- initial weight of coir pith

## Characterization of treated coir pith samples using FTIR and SEM

**SEM analysis:** SEM studies could reveal the surface topography and structure of the samples. Figure 4 shows the SEM analyses of both enzyme-treated and chemically treated samples were recorded and compared with that of intact coir pith for distinguishing any morphological changes during the treatment processes. It was observed that prolonged acetylation (> 12 hrs) leads to rupturing of the porous structure of coir pith. In activated carbon the whole polymer lattice was found ruptured. Rupturing of polymer lattice destroys the capillaries, but increases the surface area of the adsorbent.

**FT-IR analysis of the samples:** Infra red (IR) spectrum of organic molecules yield valuable information on different functional groups present in them. In the present study, the IR spectra of coir pith and various derivatives thereof were recorded using a diffuse reflectance assembly. Prior to that the sample was ground to a fine powder with dry KBr and the homogenous solid solution containing 1-2 % of the substance was used for recording IR spectra. Figure 5



Scheme 1 Reaction process

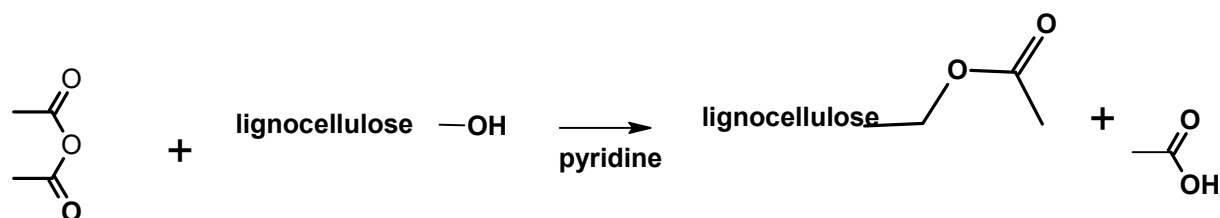


Figure 1. Mechanism of acetylation

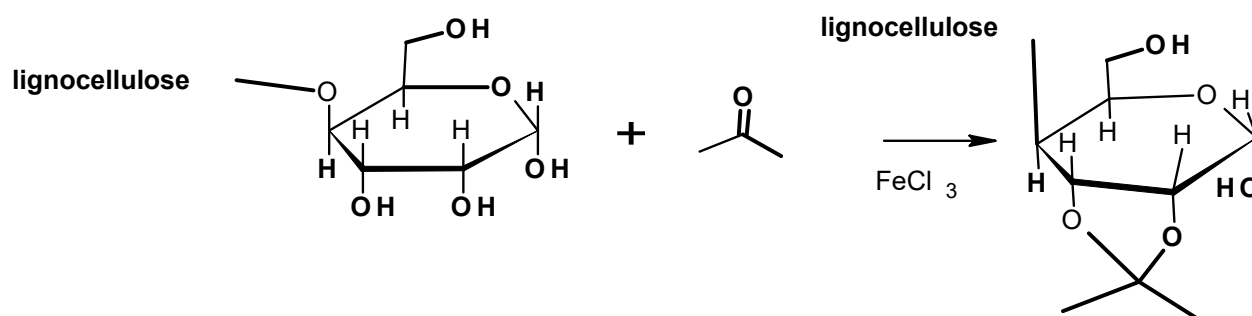


Figure 2. Reaction of coir with acetone

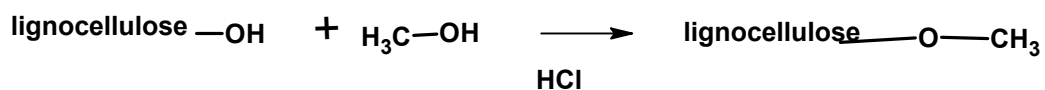


Figure 3. Process of Methanolysis

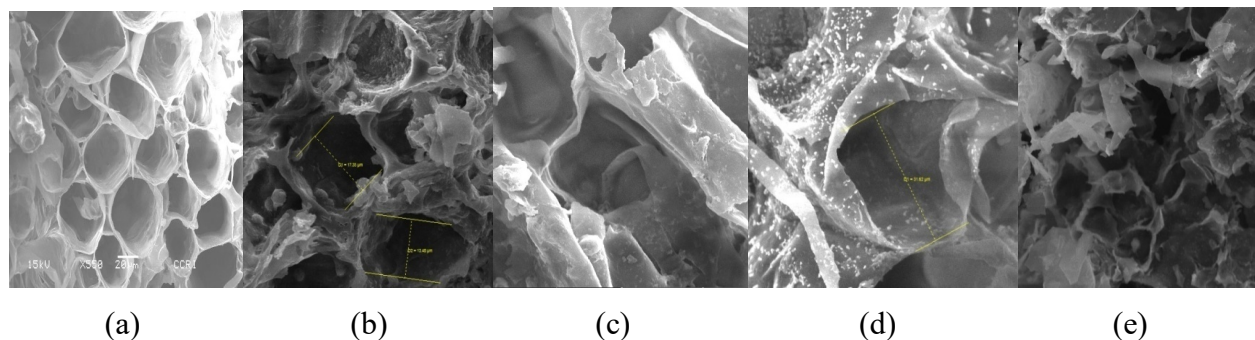


Figure 4. SEM images of (a) coir pith, (b) MIBK, (c) acetylated coir pith, (d) protease treated coir pith and (e) activated carbon

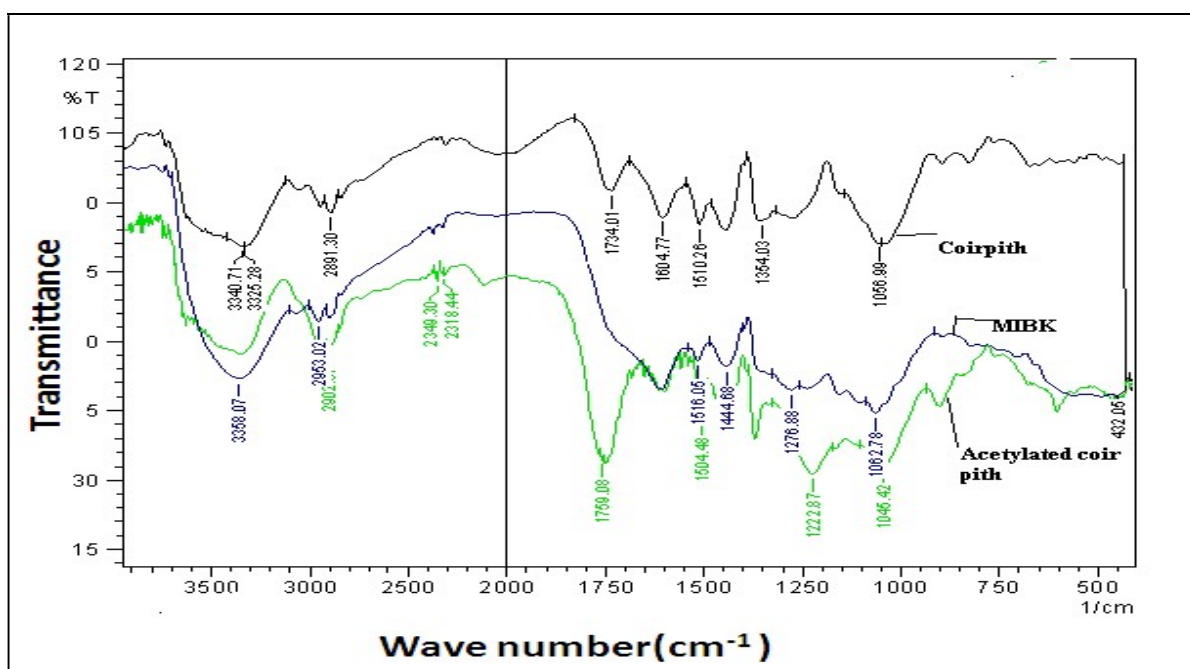


Figure 5. FTIR spectra of coir pith and its chemical derivatives

shows IR spectra of treated coir pith samples. The major peaks at 3400, 2900, 1730, 1600 & 1510  $\text{cm}^{-1}$ . The peaks in the region 2800-3000 and 3300-4000  $\text{cm}^{-1}$  arise from aliphatic C-H and O-H stretching vibrations. The values at 1510 and 1600  $\text{cm}^{-1}$  could be aromatic skeletal vibrations (of lignin), while the small absorption at 1730  $\text{cm}^{-1}$  may be due to ester groups (minor component).

Figure 6 shows the acetylated coir pith, a strong IR absorption at 1742  $\text{cm}^{-1}$  due to acetate ester moiety, as expected. This absorption increased in a linear manner up to 12 hours of reaction time, but decreased afterwards. Simultaneously, the band at 3358  $\text{cm}^{-1}$  due to O-H absorption decreased, indicating conversion of OH into OAc groups. The IR peak at 1240  $\text{cm}^{-1}$  corresponds to the C-O stretching frequency of acetyl groups. The absence of peak at 1700  $\text{cm}^{-1}$  and 1840  $\text{cm}^{-1}$  in the spectrum of acetylated samples indicates that byproduct acetic acid and unreacted reagent acetic anhydride are successfully removed from the compound.

Graph 1 displays the extent of acetylation which was determined using FTIR method from the ratio of the intensity of carbonyl (C=O) stretching band at 1740-1745  $\text{cm}^{-1}$  to that of C-O stretching absorption of the cellulose backbone at 1020-1040  $\text{cm}^{-1}$ . The value increased with reaction time up to 12 hours, but thereafter decreased, probably due to competing deacetylation reaction or collapse of capillaries or combination of both.

In the IR spectra of activated carbon (Figure 7) comprised of a peak at 2860  $\text{cm}^{-1}$  corresponds to aliphatic  $\text{CH}_2$  and  $\text{CH}_3$  stretching. Peak at 1720  $\text{cm}^{-1}$ . The Peak around 1380  $\text{cm}^{-1}$  corresponds to C-O-C stretching in carboxylic acids and esters

## Observations and Results

Coir pith with particle size of 0.5-1mm was chosen for both chemical and biological treatments since it showed higher oil adsorption as compared to those having particle size lower than 0.5 mm (**Graph 2**). Among the different enzyme treated samples, protease treated coir

pith displayed highest oil adsorption efficiency (**Graph 3, 4 & 5**).

Comparison of oil adsorption efficiency of chemically treated coir pith samples indicated the acetylated coir pith to display highest oil adsorption efficiency, followed by activated carbon (**Graph 6 & 7**). The other samples (methylated and ketal derivatives) did not show any noticeable improvement in oil adsorption efficiency, perhaps due to lower product yields. In order to obtain the optimum reaction conditions for acetylation, the reaction was repeated for varying durations. It was noticed that the highest oil adsorption efficiency was shown by the sample acetylated for 12 hours. Further increase of reaction time led to a reduction in oil adsorption (**Graph 8**). IR spectroscopy and chemical methods revealed maximum number of acetate groups to be present in coir pith acetylated for 12 hours. While the values steadily increased up to 12 hours, reaction carried out beyond this time led to decrease in acetate content as well as reduction in oil adsorption capacity (**Graph 7 & Figure 6**). The reduced oil adsorption and acetate content in samples treated beyond 12 hours may be due to (1) partial deacetylation due to hydrolysis /or cyclic acetal formation and (2) destruction of fine capillaries within the coir pith due to prolonged exposure to acetic acid / acetic anhydride. The process of deacetylation is depicted in (Scheme 1 & 2).

The activated carbon was characterized by the physical properties pH, particle density, bulk density and porosity (**Graph 9, 10 & 11**).

The granules of coir pith can be spread on the polluted area. Coir pith has higher affinity for oil and form foam - like layer after the absorption of oil. The floating layer of coir pith containing oil can be easily scooped off. Absorbed oil is conveniently recovered from the sample by mechanical squeezing and can be repeatedly used for additional oil cleanups.

The liberation of water-soluble, colored poly phenols from coir pith soaked in water is a serious drawback, preventing its wider usage as an oil spill cleaner. Our studies indicate that amount of polyphenols leaching from acetylated



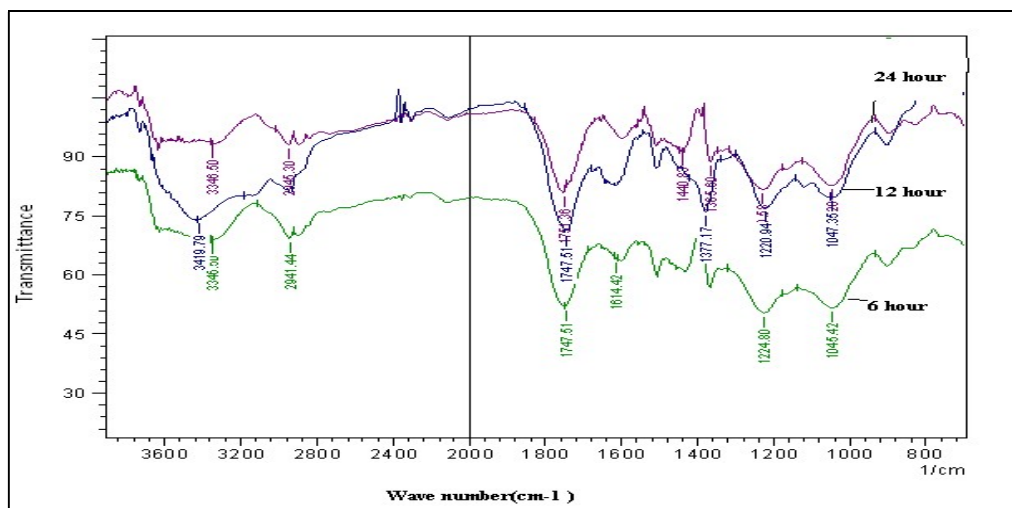


Figure 6. FTIR spectra of Comparison of extent of acetylation reaction

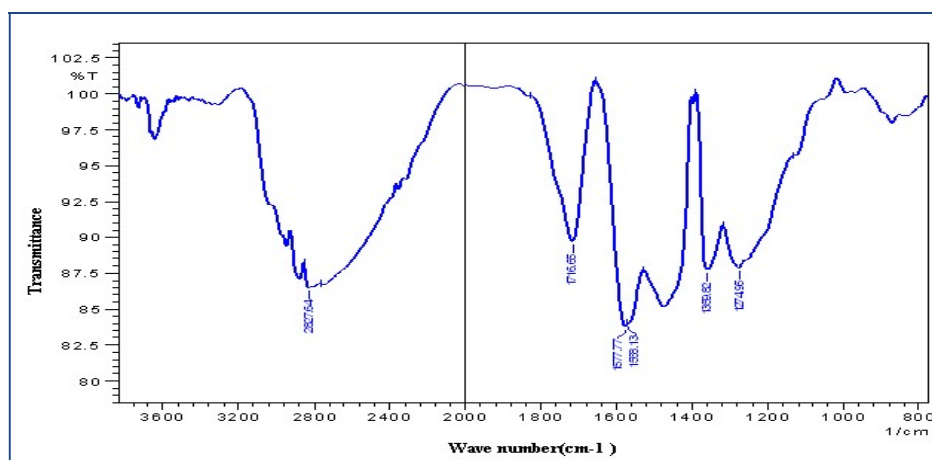
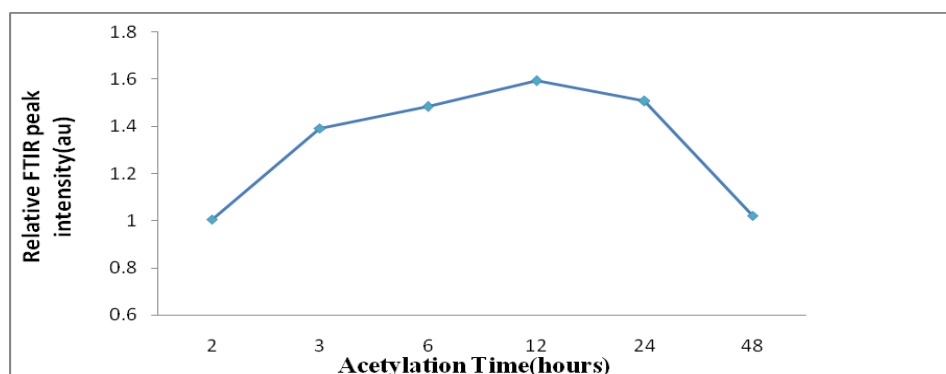
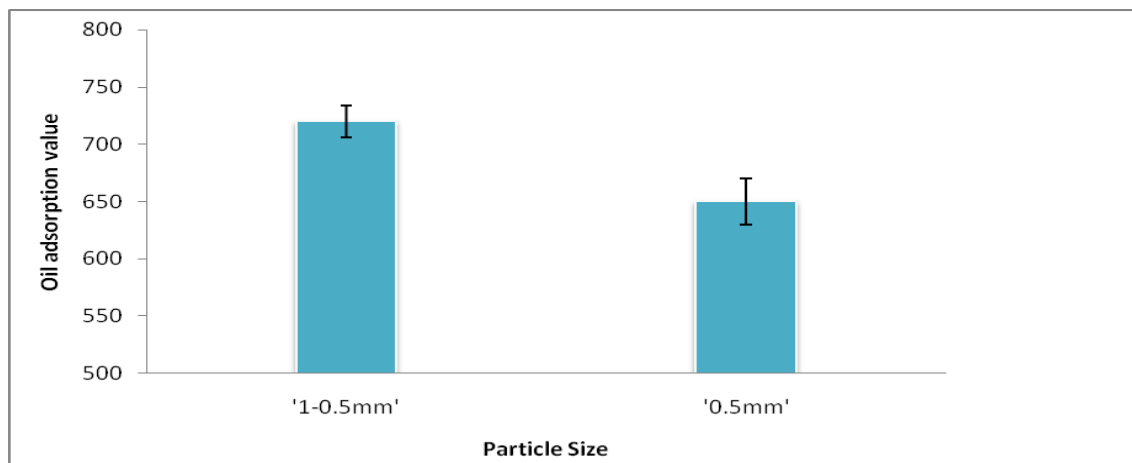


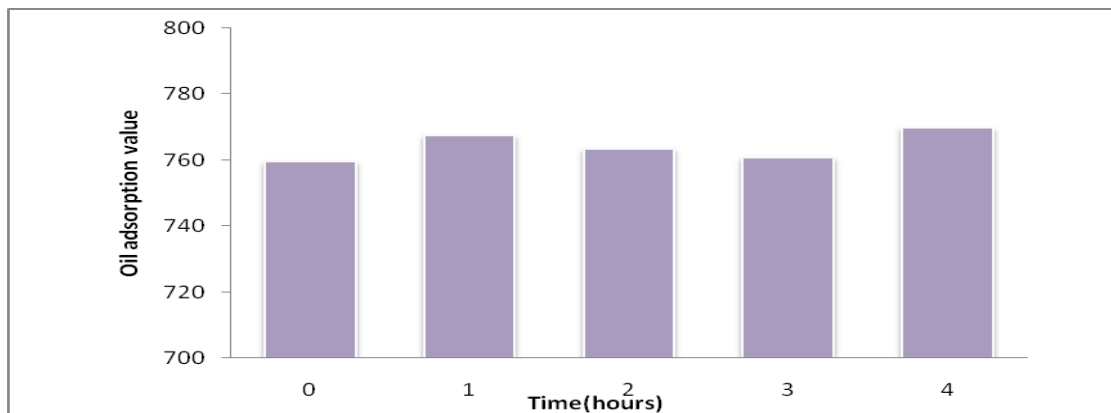
Figure 7. FTIR spectra of activated carbon from coir pith



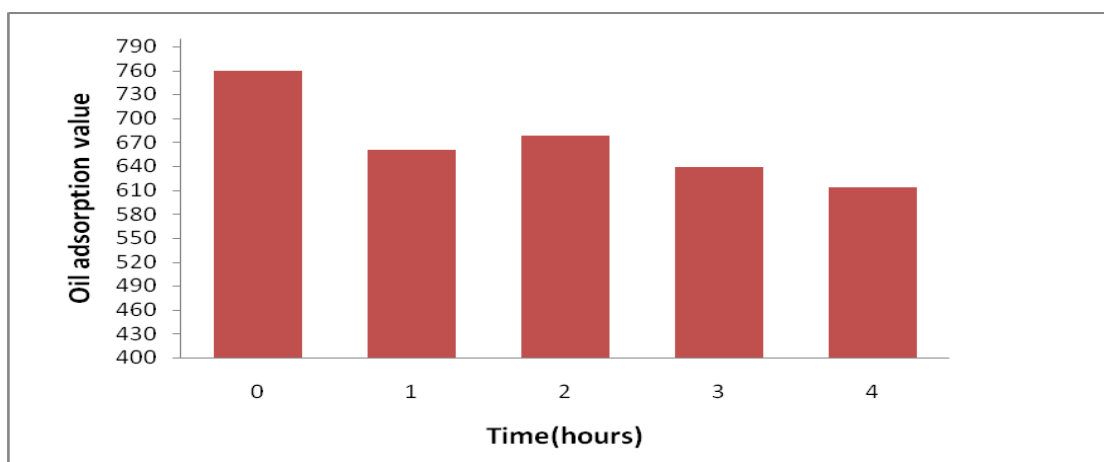
Graph 1. Comparison of the relative peak intensity of the acetylated products



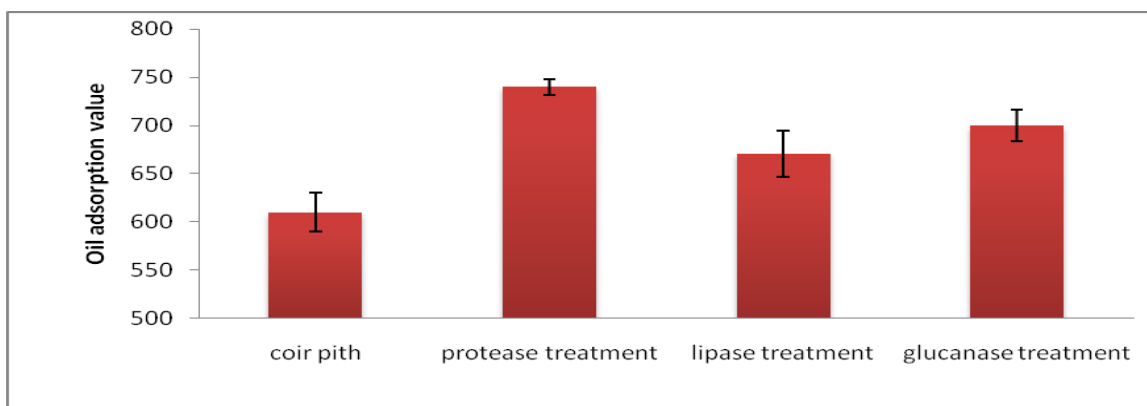
**Graph 2. Effect of particle size on the oil adsorption capacity of the coir pith**



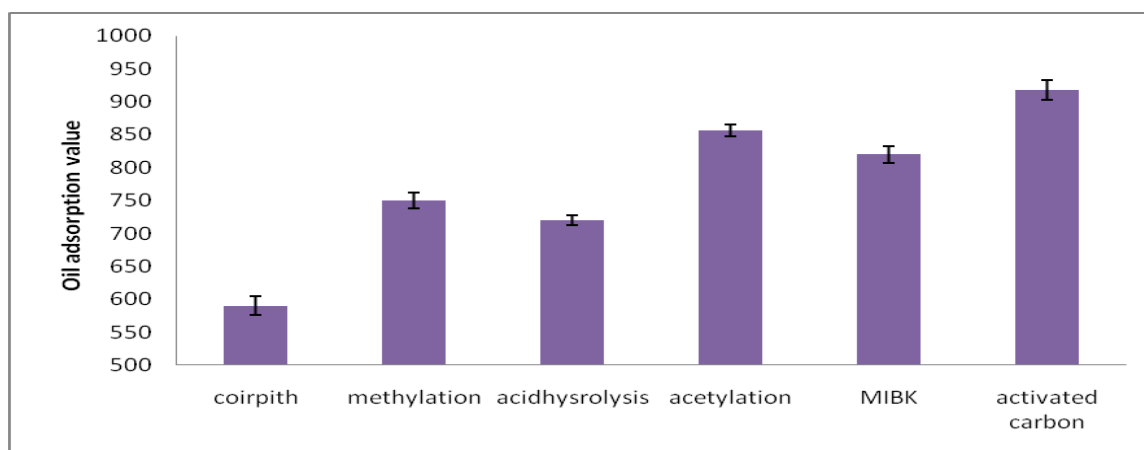
**Graph 3. Comparison of oil adsorption efficiency of protease treated samples**



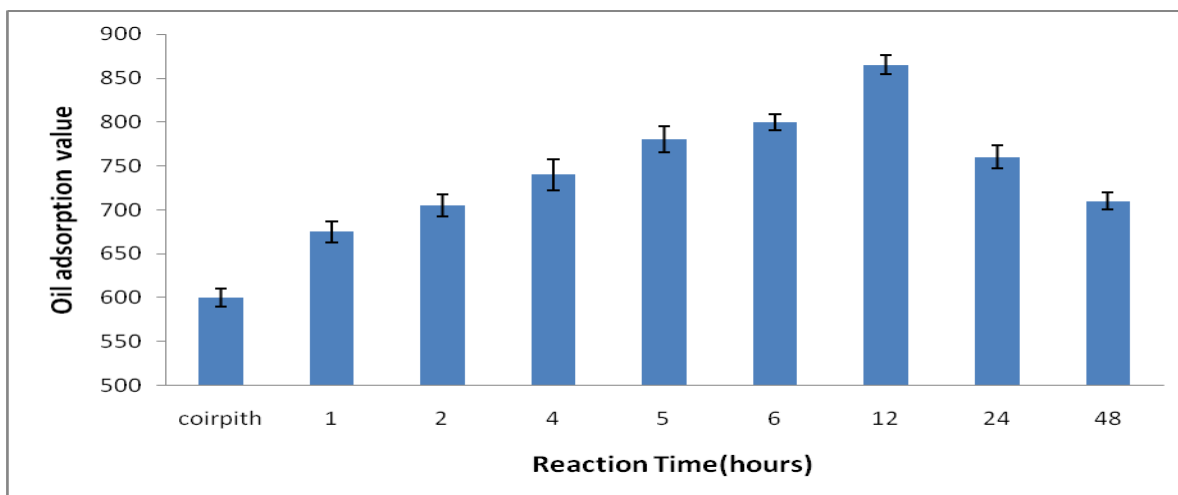
**Graph 4. Comparison of oil adsorption efficiency of lipase treated samples**



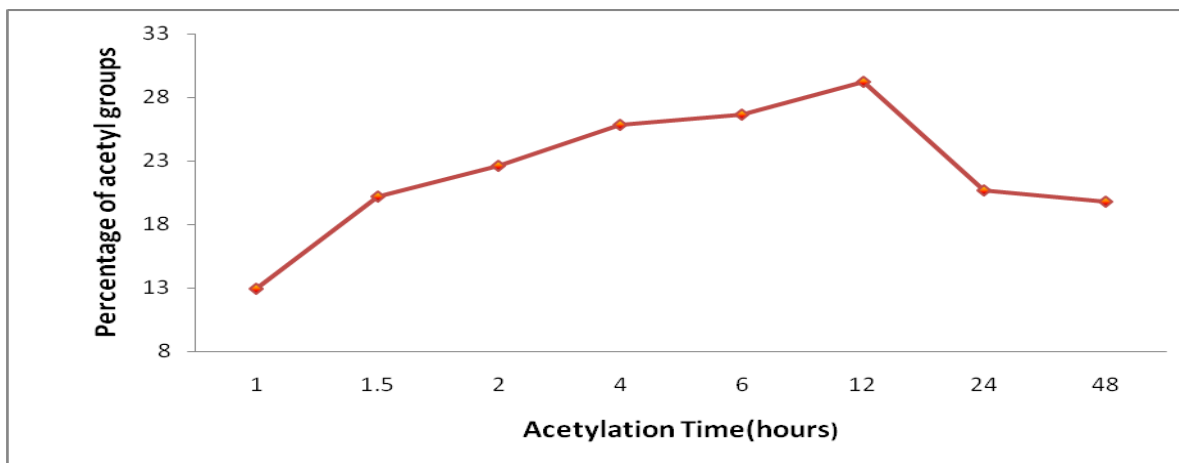
**Graph 5. Comparison of oil adsorption efficiency by coir pith after different enzyme treatments**



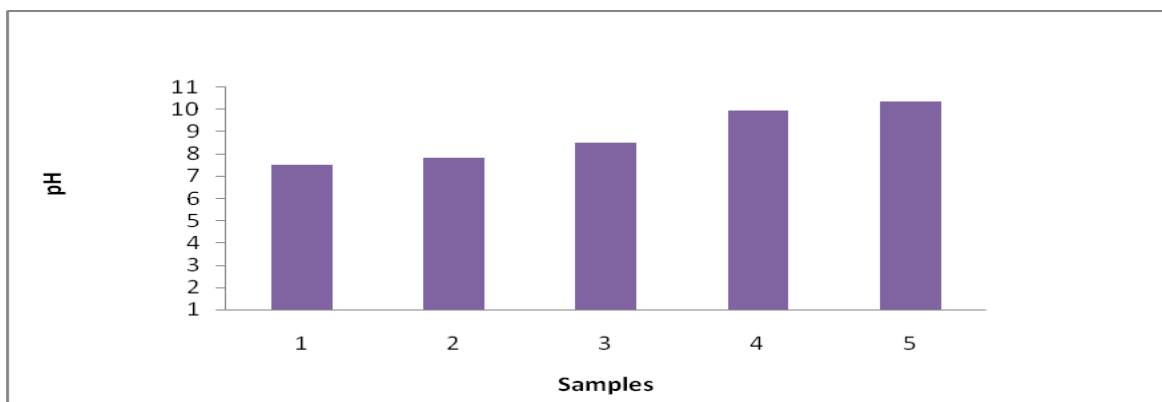
**Graph 6. Comparison of the oil adsorption efficiency of chemically treated samples**



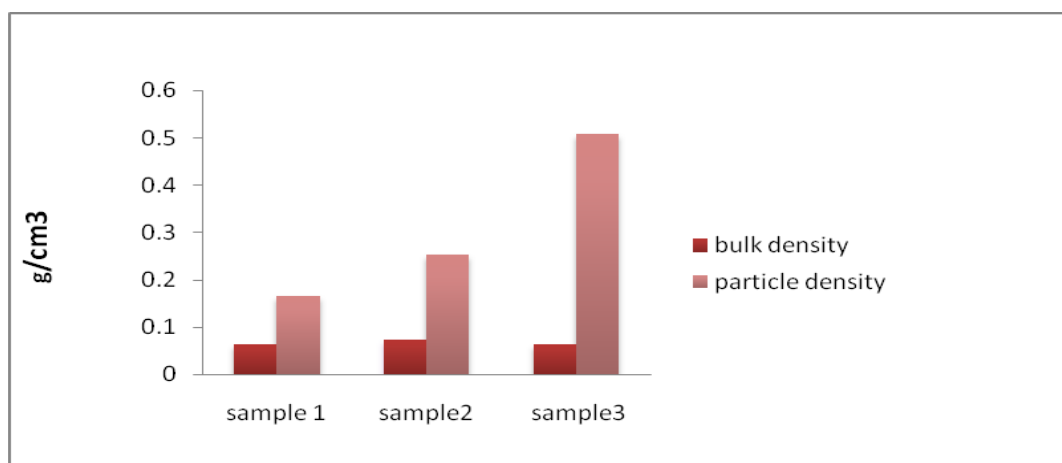
**Graph 7. Comparison of the oil adsorption efficiency of different acetylated products**



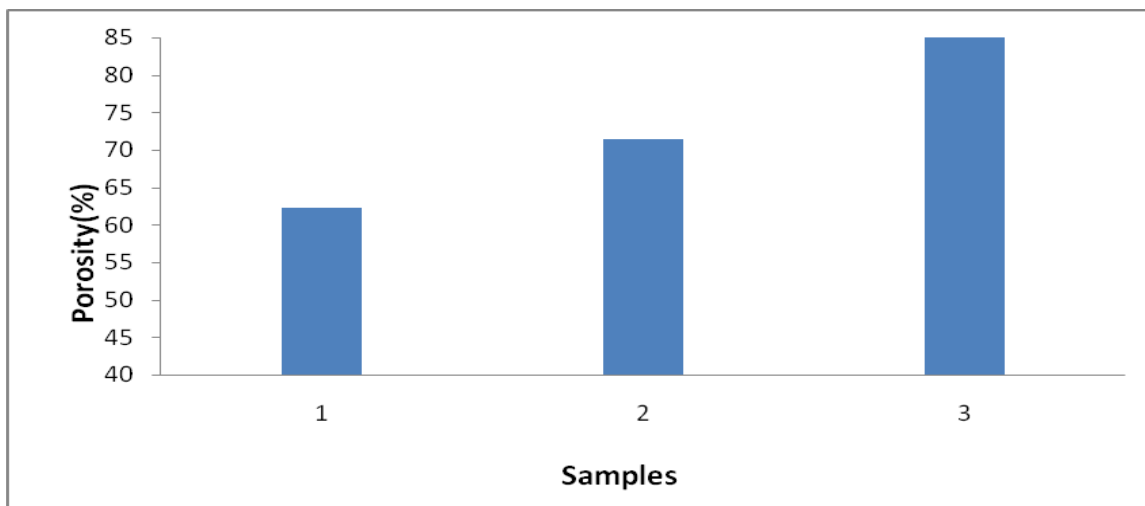
**Graph 8. Estimation of percentage of acetyl groups in different acetylated samples**



**Graph 9. pH of activated Carbon**



**Graph 10. Particle density and bulk density of the activated carbon**



Graph 11. Porosity of the activated carbon samples

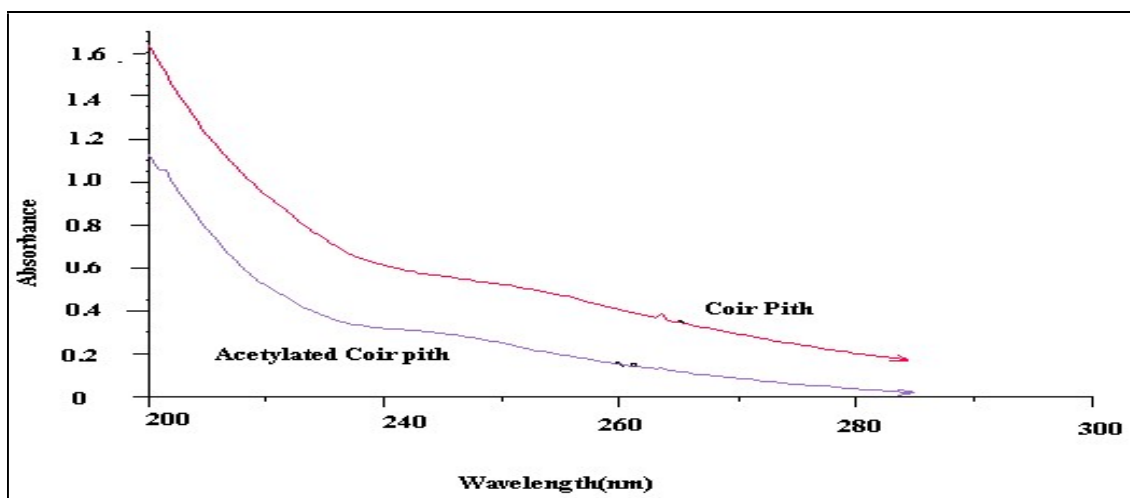
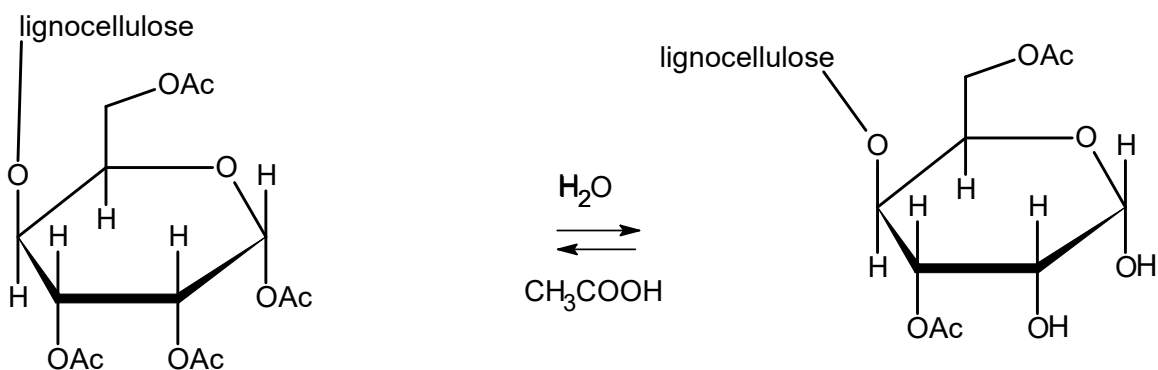
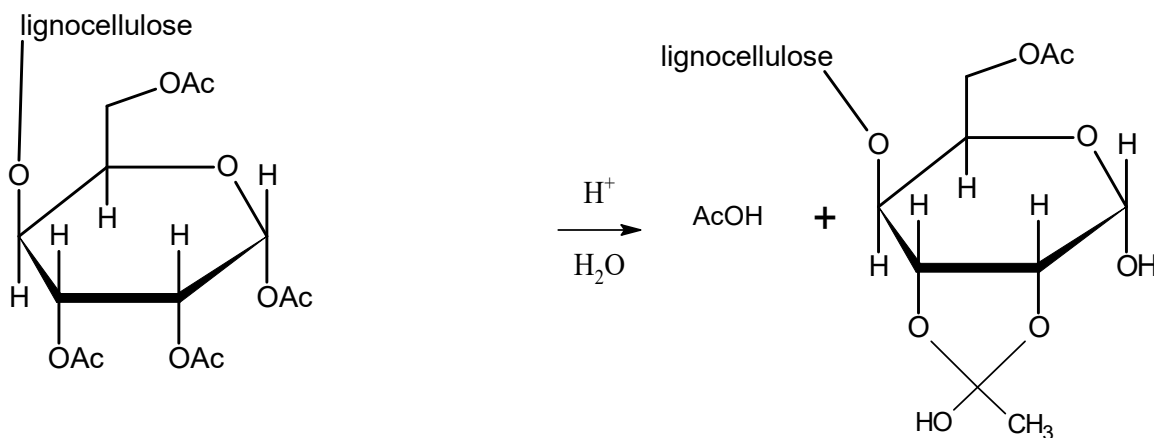


Figure 8. UV-Vis spectra of water samples after soaking Coir pith and acetylated coir pith for one week each



Reaction scheme 1. Deacetylation mechanism



**Reaction Scheme 2. Deacetylation involving cyclic structure**

coir pith is much lower than natural coir pith (Figure 8).

It is estimated that cost of production of 50g of acetylated coir pith using GR Grade acetic anhydride is around Rs 750.00. Efficient recovery of unreacted acetic anhydride and acetic acid, the reaction byproduct will reduce this cost further.

### Conclusion

Coir pith, a byproduct of coir industry and abundantly available raw material can be converted into a value added product of potential application in environment protection. Among the different biological (enzymatic) and chemical methods tested, chemical modification involving acetylation of coir pith was found to adsorb maximum oil. About 50% enhancement in oil adsorption by acetylated coir pith, treated for 12 hours was observed. Beyond 12 hours, the integrity of coir pith was observed to be lost due to physical crumbling and deacetylation as revealed through SEM, FT-IR and chemical methods (saponification). Acetylated sample is also more environment-friendly, due to lower leaching of poly phenols into the environment as compared to untreated coir pith and therefore the potential of coir pith can be harnessed for mopping up oil spills in water bodies and save the toxic effects to the marine life in aquatic environments.

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## Differential Scanning Calorimetric Analysis of Virgin Coconut Oil, Palm Olein, and their Adulterated Blends

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### Abstract

Virgin coconut oil (VCO) is a premium product with a high market value. Its authenticity and quality assurance are important to safeguard consumers from fraudulent practices. The aim of this study was to investigate the effect of adulteration by palm olein (PO) on differential scanning calorimetric (DSC) heating and cooling profiles of VCO. Pure samples of VCO, PO and their adulterated blends (5 to 30%, w/w) were subjected to thermal analysis by DSC according to a specified temperature program. DSC thermal analysis system software and SAS statistical system were used subsequently to analyze thermal data. Both cooling and heating curves of VCO were found to be vivid for fingerprint comparison of qualitative identification at 5% level of adulteration.

**Key words:** Authentication, adulteration, DSC, virgin coconut oil, thermal analysis

### Introduction

Virgin coconut oil (VCO) is a premium product extracted from fully matured coconuts using mechanical oil expellers operating under low-temperature condition (Bawalan and Chapman, 2006). Since the oil is extracted hygienically under mild temperature conditions, it looks colorless, limpid, and free from cloudiness coming from any dirt. According to previous reports, many VCO samples were found to contain very low amounts of moisture (<0.1) and free fatty acid (<0.1). Other than these, the levels of hydro peroxides present in them were negligibly small to be detected (Jayasundera, *et al* 2003). The growing demand for VCO is mainly due to its health attributes, which arise from its triacylglycerols composition that contain considerable amounts of medium-chain fatty acids (Nevin and Rajamohan, 2004). Medium-chain triglycerides (MCT) are generally considered to be healthy since they are biologically inert source of energy that the human body finds reasonably easy to metabolize. Owing to this reason, medium-chain triglycerides have applications in treatments of a variety of malabsorption ailments (Nagao and Yanagita, 2010). Apart from the benefits on human nutrition, VCO is also known for its potential applications in hair oils, baby oils and massage oils used in aroma therapies. All these have caused VCO to catch up unmatched demand in the market place; this makes the oil to be vulnerable for adulteration with less expensive oils such palm olein. Palm olein, the liquid fraction of palm, is relatively cheaper to purchase due to its low cost of production. This type of economic fraud will alter the chemical composition of VCO affecting its nutritional and therapeutic values.

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Authentication of VCO against adulteration practices has been a challenging task for many researchers in the past. For instance, detection of VCO adulteration by palm kernel oil remained as a challenge due to their close similarities in fatty acid and TAG compositions. Despite this, various analytical approaches adopted by researchers helped to provide solutions to adulteration issues (Xu *et al.*, 2011; Marina *et al.*, 2009; Manaf *et al.*, 2007). Differential scanning calorimetry has been one of the several instrumental techniques used by researchers for authentication of various oils and fats. As DSC is an analytical tool that can provide well-defined curves for unadulterated neat oils, it helped to give fingerprints for authentication as well as quality assurance purposes. In a pioneering effort, Dyzel and Baish (1992) demonstrated the use of DSC in the identification of various edible vegetable oils. This study triggered the curiosity of several researchers to investigate the use DSC to detect adulterations in several cases (Marikkar, 2015; Marina *et al.*, 2009). In majority of the studies, DSC provided both cooling and heating curves that gave plenty of information about thermal transitions and associated thermal parameters to compare an authentic VCO sample with adulterated ones. Most of the time, deviations in the DSC heating and cooling profiles of suspected samples enabled the researchers to detect adulterations. Nevertheless, there has been a knowledge gap in the literature about DSC thermodynamic parameters that show high-sensitivity to low-levels of adulteration by palm olein. This can be determined through the use of statistical correlation between different DSC parameters and varying levels of adulterations in VCO.

### Materials and Methods

**Materials:** Six samples of VCO produced from 10 to 11 month old nuts were obtained from Malaysian Agricultural Research and Development Institute, Selangor, Malaysia. Six samples of PO were purchased from Lam Soon Edible Oils Sdn. Bhd, Selangor, Malaysia. All chemicals used in this study were of analytical grade unless otherwise specified (Sigma-Aldrich). For quantitative analysis, samples of

virgin coconut oils with varying levels of adulterant oil were prepared as given below.

**Blend preparation:** VCO and adulterant (PO) were mixed together in differing ratios to form a set of samples. A total of six samples were prepared: (A<sub>1</sub>) 95:5, (A<sub>2</sub>) 90:10, (A<sub>3</sub>) 85:15, (A<sub>4</sub>) 80:20, (A<sub>5</sub>) 75:25, (A<sub>6</sub>) 70:30 (w/w) and identified by the mass ratio of VCO to PO.

**DSC thermal analysis:** DSC analysis was performed by using Mettler Toledo DSC, Model 823 (Mettler-Toledo GmbH, Zurich, Switzerland) equipped with thermal analysis STAR<sup>c</sup> system. Nitrogen (99.9% purity) was used as the purge gas rate at a rate of 20mL/ min. Approximately 6-8mg of oil sample was weighed and placed in a standard DSC aluminium pan and then hermetically sealed. An empty covered DSC aluminium pan was used as a reference. In order to obtain cooling and melting profile the following temperature program was set: 70°C isotherm for 5min, cooled at 5°C/ min to -70°C and held for 5min. The same rate was used to heat the sample from -70°C to 70°C. The thermal data was analyzed using DSC software library program (Marikkar *et al.*, 2013).

**Statistical analysis:** The relationships between each DSC parameter and adulteration level were determined by Pearson's correlation analysis. Statistical significance was declared at 0.05 probability level. The DSC parameters and level of adulteration were analyzed using a stepwise procedure in SAS to develop prediction models for quantification (SAS 1998). The significance level of an independent variable to enter and stay in the calibration mode was set to 0.05 during execution of the stepwise variable selection in SAS procedure "REG".

### Results and Discussions

DSC analysis of lipids is generally performed under two different conditions, namely cooling process and heating process, which give cooling curve and a melting curve, respectively. While cooling process provides details of the thermal events associated with crystallization, heating process provides information about the thermal events associated

with melting. According to previous studies, DSC curves of most edible oils and fats are strongly influenced by their fatty acid and TAG compositions (Marikkar, 2015; Dyzel and Baish, 1992). Because of this reason, changes in lipid composition due to adulteration might cause changes in both cooling and heating curves. This has provided a scientific basis for detection of adulterations in edible oils, butter, cheese, etc.

### **DSC Heating curves of VCO**

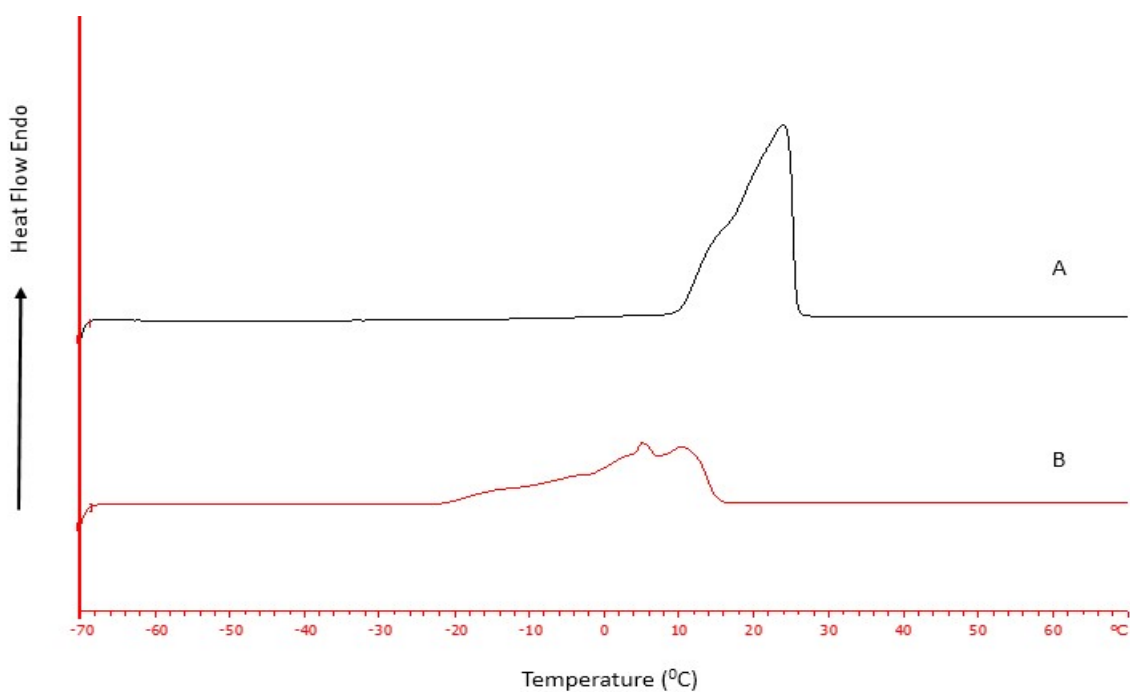
The overlay of DSC curves presented in Figure 1 compares the DSC heating curves of VCO (Curve-A) and PO (Curve-B). Based on the illustration, DSC heating curves of VCO and PO were significantly different; VCO displayed one major exothermic peak at 23.78 °C with a shoulder peak at 14.20 °C. The existence of the shoulder peak close to the major endothermic peak is attributed to the co-melting of two different groups of TAG molecules. While the smaller shoulder peak indicates the lower melting TAG components of VCO, the major peak indicates the higher melting TAG components. The overlay of DSC curves presented in Figure 2 showed the gradual deviations in DSC features caused by adulteration. The changing proportion of adulterant affected all DSC parameters of the two peaks. As discussed before by previous researchers, the changing pattern of thermal profiles of the samples especially in peak 1 (major peak) and peak 2 (shoulder peak) could be attributed to the TAG compositional changes caused by adulteration. With the increasing percentage of PO, the shoulder peak started to flatten forming a clear separation from the major peak. In Peak-1 (the major peak), there was remarkable shift in the peak temperature from 23.78 to 19.09°C. The reduction of its peak area (A) from 426.8 to 298.4 mJ, the decrease in the onset temperature (O) from 16.75 to 14.25°C and the decline of the peak height (H) from 7.16 to 4.4 mW caused a reduction in the sharpness of the peak (Table 1). In Peak-2 (the shoulder peak), there was remarkable shift in the peak temperature from 14.20 to 9.20°C while the peak area (A) has increased from 11.34 to 42.38 mJ. Apart from these, the decreases in onset

temperature (O) from 9.57 to 4.43°C and the peak height (H) from 1.64 to 0.41 mW caused reduction in the sharpness of the peak.

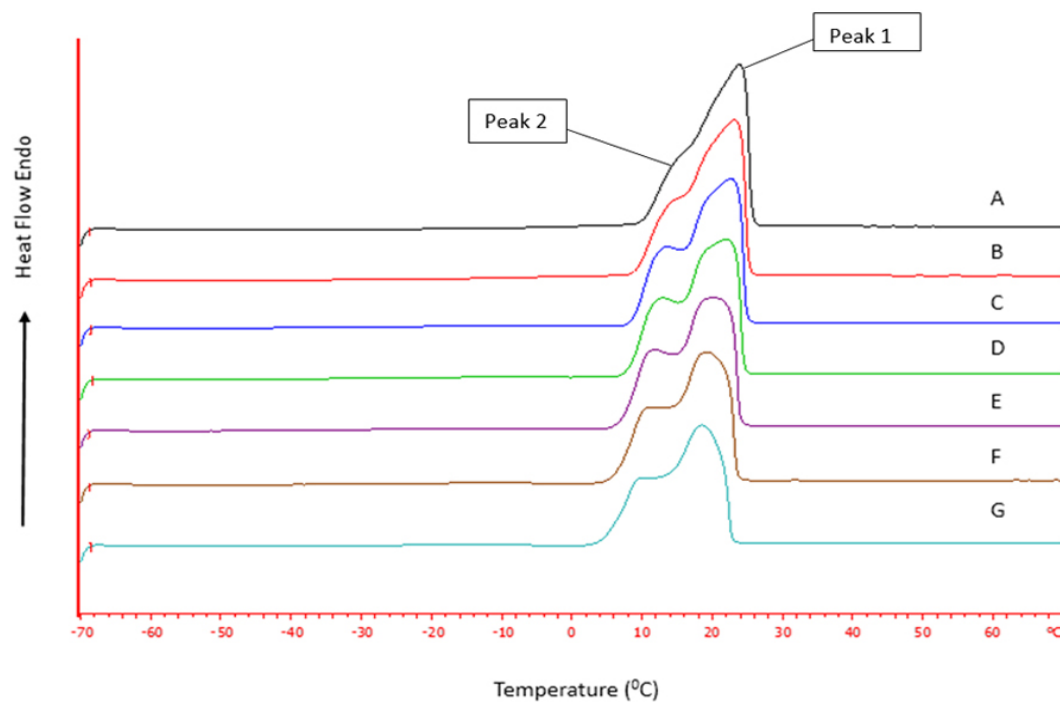
The linearities between individual DSC parameter and the varying level of the adulterant are compared as shown in Table 1. Pearson correlation analysis showed that all four DSC parameters of peak-1 namely, A, T, H, and O displayed good correlation. However, for peak-2, the only DSC parameter showing good correlation was T (Table 1). When each of the DSC parameter of peak-1 was subjected to stepwise multiple regression analysis for quantification purpose (Table 2), the best regression model obtained was  $Y = -0.161 \text{ Temperature} + 14.0$  [ $R^2=0.993$  ( $p<0.0001$ ) and  $SE=0.17$ ]. This predictive model not only give high correlation coefficient with good confidence limit but also yielded a low value for standard error (SE). According to Table 2, other regression models obtained using peak onset temperature, peak area and peak height were found to display weaker correlation.

### **DSC Cooling curves of VCO**

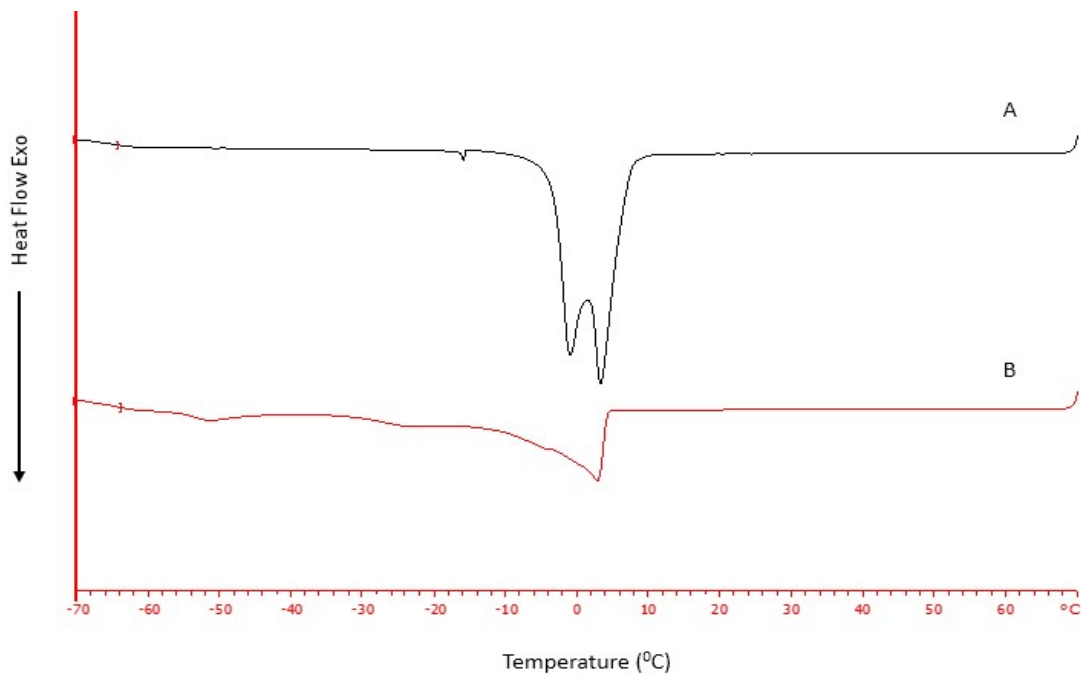
The overlay of DSC curves presented in Figure 3 compares the DSC cooling curves of VCO (Curve-A) and PO (Curve-B). Based on the illustration, DSC heating curves of VCO and PO were significantly different; VCO displayed two overlapping exothermic peaks at 3.56°C and -0.89°C. The occurrence of two overlapping exothermic peaks was the direct result of co-crystallization of TAG molecules, which were closely similar in their melting temperatures. Based on the illustration in Figure 4, addition of PO to VCO caused the two overlapping peaks to undergo morphological changes in their thermal behavior. As a notable feature, no new peak correspond to the presence of adulterant was appeared. When percentage of PO increased in the admixture, there was a slight shift in the peak temperature of peak-1 (the major peak) from 3.56 to 1.95°C and increase in the peak area (A) from 112.47 to 190.23 mJ. Simultaneously, the onset temperature (O) tended to decrease from 5.93 to 2.67°C while the peak height (H) went up from 4.89 to 10.31 mW (Table 1). In Peak-2, there was a shift in peak temperature (T) from –



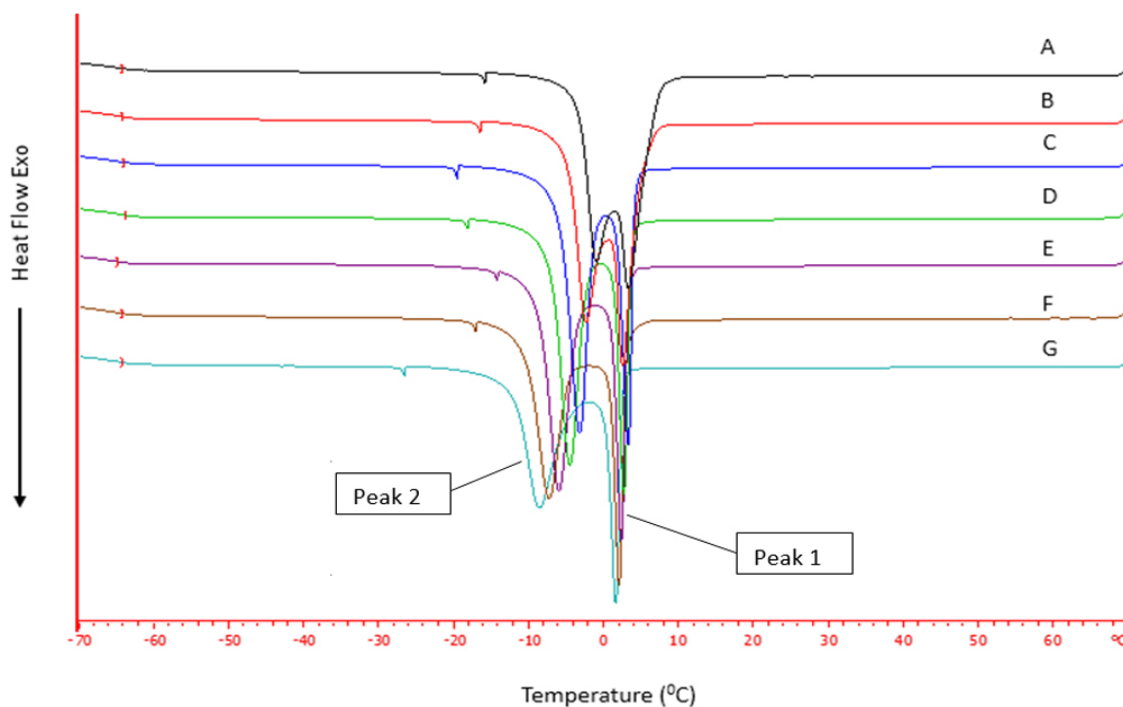
**Figure 1. DSC heating curves of (A) virgin coconut oil (VCO) and (B) palm olein (PO)**



**Figure 2. DSC heating curves of (A) virgin coconut oil (VCO), (B) VCO adulterated with 5% palm olein (PO), (C) 10% PO, (D) 15% PO, (E) 20% PO, (F) 25% PO, and (G) 30% PO**



**Figure 3.** DSC cooling curves of (A) virgin coconut oil (VCO) and (B) palm olein (PO)



**Figure 4.** DSC cooling curves of (A) virgin coconut oil (VCO), (B) VCO adulterated with 5% palm olein (PO), (C) 10% PO, (D) 15% PO, (E) 20% PO, (F) 25% PO, and (G) 30% PO

**Table 1. Pearson correlation coefficient between % level of adulterant and each of DSC parameter associated with peaks in the cooling and heating curves<sup>1</sup>**

Curve Type	Peak	DSC Parameter	DSC Value Range	Correlation Coefficient
Heating	Peak 1	A	298.39 to 426.88	-0.947(p< 0.0001)
		T	19.09 to 23.78	-0.977(p< 0.0001)
		H	4.4 to 7.16	-0.996(p< 0.0001)
		O	14.25 to 16.75	-0.979(p< 0.0001)
	Peak 2	A	11.35 to 42.38	+0.505(p< 0.248)
		T	9.2 to 14.2	-0.996(p< 0.0001)
		H	0.41 to 1.64	+0.524(p< 0.228)
		O	4.43 to 9.57	-0.871(p< 0.011)
Cooling	Peak 1	A	-190.23 to -112.47	+0.337(p< 0.144)
		T	1.95 to 3.56	-0.898(p< 0.006)
		H	4.89 to 10.31	+0.701(p< 0.079)
		O	2.67 to 5.93	-0.976(p< 0.0001)
	Peak 2	A	-300.58 to -67.44	-0.644(p< 0.118)
		T	-8.52 to -0.89	-0.999(p< 0.0001)
		H	4.4 to 11.55	+0.217(p< 0.639)
		O	-5.23 to 0.99	-0.998(p< 0.0001)

<sup>1</sup>Abbreviation: A, peak area; T, peak temperature; H, peak height; O, peak onset temperature

**Table 2. Summary of stepwise regression analysis carried for predictive model of PO content using DSC parameters of the heating curve<sup>1</sup>**

Model	Regression equation	R <sup>2</sup>	SE
Peak 1			
1	Y = - 0.161 Temperature + 14.0	0.993(p< 0.0001)	0.17
2	Y = 0.905 Area + 33.9	0.255 (p< 0.042)	18.32
3	Y = 0.021 Height + 0.828	0.274 (p< 0.030)	0.40
4	Y = - 0.151 Onset + 10.5	0.759 (p< 0.0001)	1.00
Peak 2			
1	Y = - 0.153 Temperature + 24.1	0.995 (p< 0.0001)	0.39
2	Y = - 3.80 Area + 4.36	0.896 (p < 0.0001)	15.32
3	Y = - 0.0952 Height + 7.22	0.992 (p< 0.0001)	0.10
4	Y = - 0.0847 Onset + 17.0	0.957 ( p< 0.0001)	0.21

<sup>1</sup>Abbreviation: Y, % of adulterant; SE, standard error**Table 3. Summary of stepwise regression analysis carried for predictive model of PO content using DSC parameters of the cooling curve<sup>1</sup>**

Model	Regression equation	R <sup>2</sup>	SE
Peak 1			
1	Y = - 0.0485 Temperature + 3.58	0.806 (p< 0.0001)	0.28
2	Y = - 2.24 Area - 1.57	0.375 (p< 0.001)	34.17
3	Y = 0.191 Height + 7.23	0.491 (p< 0.006)	2.30
4	Y = - 0.102 Onset + 5.52	0.953 (p< 0.0001)	0.27
Peak 2			
1	Y = - 0.253 Temperature - 0.771	0.997 (p< 0.001)	0.15
2	Y = - 7.97 Area - 152	0.415 (p< 0.103)	111.95
3	Y = 0.055 Height + 6.89	0.047 ( p< 0.019)	2.95
4	Y = - 0.214 Onset + 0.951	0.995 (p< 0.0001)	0.17

Abbreviation: Y, % of adulterant; SE, standard error

0.89 to -8.52 while the peak area (A) increased from -67.44 to -300.58 mJ. In the meantime, onset temperature (O) of this peak tended to decrease in value from 0.99 to -5.23°C while the peak height (H) fell from 4.4 to 6.02 mW leading to the reduction in the sharpness of the peak (Table 1).

Pearson correlation analysis showed that among the DSC parameters of peak-1, peak onset temperature (O) displayed good correlation [-0.976;  $p < 0.0001$ ] while in peak-2, both peak temperature T [-0.999;  $p < 0.000$ ] and peak onset O [-0.998;  $p < 0.0001$ ] were found to display good correlations. When each of the DSC parameter of peak-1 was subjected to stepwise multiple regression analysis for quantification purpose (Table 3),  $Y = -0.102 \text{ Onset} + 5.52$  [ $R^2 = 0.953$  ( $p < 0.0001$ ) and  $SE = 0.27$ ] was obtained as the best regression model. This predictive model not only give high correlation coefficient with good confidence limit but also a very low SE value. When the same procedure was repeated for DSC parameters associated with peak-2, the following two regression models having high correlation coefficient with good confidence limit and very low SE values were obtained:

$$Y = -0.214 \text{ Onset} + 0.951 \quad [R^2 = 0.995 \quad (p < 0.0001) \text{ and } SE = 0.17]$$

$$Y = -0.253 \text{ Temperature} - 0.771 \quad [R^2 = 0.997 \quad (p < 0.001) \text{ and } SE = 0.15]$$

According to Table 3, other regression models obtained using peak area and peak heights were found to display weaker correlation.

### Conclusions

This study investigated the use of DSC thermal analysis for detecting adulteration in VCO by PO. Comparative DSC curves of both heating and cooling processes of VCO and PO were significantly different. Owing to this reason, DSC curves of VCO were found to deviate remarkably after adulteration with PO. It is believed that the changing proportion of saturated to unsaturated TAG molecular ratio caused these significant changes in thermal transitions and the associated DSC parameters of

VCO. According to Pearson correlational analysis, DSC parameters namely peak temperature, onset, and peak height were sensitive parameters based on the heating curves while peak temperature, onset, and peak height was the most sensitive parameters based on the cooling curves. For quantitative estimation of adulteration levels, predictive models based on DSC parameters namely onset and peak temperature associated with cooling curve were found to have high correlation coefficient with lower standard error values.

### Acknowledgments

Authors gratefully acknowledge financial support and research facilities given by Universiti Putra Malaysia.

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## Effect of Different Weed Management Strategies on Population Changing Pattern of *Pennisetum polystachion* in Coconut Plantations of Sri Lanka

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### Abstract

*Pennisetum polystachion* is a major problematic monocotyledonous weed species and a perennial problem in intermediate zone of coconut plantations in Sri Lanka. This study was carried out to evaluate the impacts of different management systems on *P. polystachion* seedling emergence patterns. The tested treatments were application of glyphosate (T<sub>1</sub>), cover cropping with *Pueraria phaseoloides* (T<sub>2</sub>), tractor harrowing (T<sub>3</sub>), tractor slashing (T<sub>4</sub>) and tractor ploughing (T<sub>5</sub>). All the treatments were applied twice a year except T<sub>2</sub>. As T<sub>2</sub> cover crop at the initiation of the experiment and over grown conditions were managed by harrowing once a year. Based on the reduction in weed biomass, cover cropping (T<sub>2</sub>) was the best to reduce the *P. polystachion* population and to reduce *P. polystachion* seedling emergence density in the field. Chemical weeding was the second-best method to control the *P. polystachion* population in the field. The effectiveness of slashing in reducing weed seedling emergence density was lower than cover cropping and chemical weeding methods. The weed seedling emergence densities were almost similar in ploughed and harrowed plots. The seed depth of emerged seedling was very high in harrowed and ploughed treatments when compared to other treatments. Results given by T<sub>3</sub> and T<sub>5</sub> indicates that loosening the soil creates more favorable environment for the germination of weed seeds buried in soil. Therefore, it can be argued that the elimination of weed seeds in the top 2cm or 4cm in the soil seed bank by any means is likely to reduce the level of weed infestation by about 60% to 95%. Results also indicated that burying rhizomes in ploughing and harrowing treatment plots at the depths below 30 - 40 cm is effective in controlling germination of this weed species. This experiment also suggested that keeping rhizomes on the soil surface without burying for durations of 5 – 15 days would produce weak plants with poor development.

**Key words:** Seedling emergence, Tiller emergence, Cover crops, Harrowing, Ploughing

### Introduction

Coconut (*Cocos nucifera* L) is the most extensively cultivated major plantation crop in Sri Lanka. It is a tropical perennial plantation crop and its canopy structure requires a wide spacing between palms, which permits abundant sunlight to the understory. As a result, the unutilized space beneath the plantation is invaded by a wide range of perennial and annual weed species (Liyanage and Liyanage, 1989). Such weeds invariably compete with coconut for soil moisture and nutrients, affecting growth and yield and obstructing routine management practices (Senarathne and Sangakkara,

2010). Management of the understory weed growth is, therefore, considered an essential step in maintaining the plantation. In fact, the cost of weeding (20% of the total cost of production of the plantation) accounts for a substantial proportion of the total recurrent expenditure for maintenance. Therefore, weeds in coconut plantations in Sri Lanka not only reduce yield due to crop weed competition but also add to the production cost. Therefore, there is an acute need to introduce effective and economically viable weed control strategies for coconut growers in Sri Lanka.

Rhizomatous weeds are the most difficult to control because of their vegetative reproduction by underground propagules (Holt and Orcutt, 1996). *Panicum maximum*, *Panicum repens*, *Imperata cylindrica*, *Pennisetum polystachion* and *Cyperus rotundus* are five rhizomatous grass weeds that exist and problematic in the coconut lands in Sri Lanka (Senarathne *et al.*, 2003). *P. polystachion* grows in very dense stands, especially in dry regions (Smith, 1979). Reproduction occurs from rhizomes and seeds (Parsons and Cuthbertson, 1992; Smith, 1995). *Pennisetum polystachion* is a tufted annual grass with culms slender to moderately stout. Grows up to 2 m tall, usually 1-2 m. Simple or few-branched, blades 5 - 40 cm long, 5 - 18 mm wide, glabrous or pubescent. Spikes are dense, yellow brown, 5 - 25 cm long and 13-26 mm wide. Spikelets surrounded by bristles, these densely hairy at base, unequal, one longer than the others but not greatly exceeding the next one or two shorter ones and are 12 - 25 mm long. Spikelets bear 2 flowers (Stone, 1970). It is propagated by seeds and rooting cuttings. Negligence, excessive grazing, ploughing during seed disposal periods and too much exposure to sun light are some causal factors. Reproduction occurs from seeds which are light and fluffy. The seeds can be dispersed by flowing water; strong winds or they may adhere to clothing, vehicle radiators, wool and bags (Parsons and Cuthbertson, 1992; Smith, 1995; Csurhes and Edwards, 1998).

This weed grows luxuriantly in all coconut growing areas especially in the wet zone of coconut triangle in Sri Lanka. Bushes grow up to

2 to 2.5 m in height. Flowers are produced in six to eight months. November-December is the flowering season. The plant inhabits disturbed areas including roadsides, degraded pastures and waste sites (Csurhes and Edwards, 1998).

In order to develop a sustainable integrated weed management strategy to manage *Pennisetum polystachion*, a detailed understanding of the seed bank is required, incorporating germination characteristics of weed seeds, rhizomatous parts and factors that regulate emergence and establishment of seedlings in the field. Although there are many studies on weed biology, weed competition and herbicide technology, little attention has been paid to investigate the regulation of weed seedling emergence in coconut lands, which is the focus of this study. Therefore, the objective of this study was to evaluate the effect of different practices for weed management on the seedling emerging pattern, emerged seedling population and germination characteristics of rhizomes of *Pennisetum polystachion* under field condition in coconut plantations.

### Materials and methods

The study was conducted at Pallama Estate, Pallama in the low country dry zone. Agro ecological zone of this area is DL<sub>3</sub> (Punyawardena *et al.*, 2003). The area is characterized by bi-modal pattern of rain fall with an annual mean precipitation of 1200 mm. Approximately 65% of the annual rainfall is received from September to February (Maha rain season). The soil at the site is a predominantly well-drained Red Yellow Podzolic (RYP) soil with soft or hard laterite (70-90%) (De Alwis and Panabokke, 1972). Surface soil is brown in colour with a sandy loam texture. Structure development is moderate due to presence of sand in the surface soil. Sub surface soil is dark to yellowish brown in colour with prominent mottles. Reaction of the soil is strongly acidic (pH 5.0 – 5.5). Base saturation of the subsurface soil is greater than 35%. Organic carbon content in the surface soil is generally less than 1% under natural conditions (Mapa *et al.*, 2005).

The experimental design was a Randomized Complete Block design with three

replicates and plot size was four coconut squares (the spacing of the square planting system of coconut is 8.2m x 8.2m).

The treatments of the experiment were as follows.

- T<sub>1</sub>. Chemical weeding (Application of Glyphosate (N- (phosphonomethyl)-glycine) 1.44 kg a.i. per hectare)
- T<sub>2</sub>. Establishment of cover crop (*Puereriaphasioloides*)
- T<sub>3</sub>. Tractor harrowing (once in six month) (0cm - 15cm depth)
- T<sub>4</sub>. Tractor slashing (once in six month)
- T<sub>5</sub>. Tractor ploughing (once in six months) (0cm - 45cm depth)
- T<sub>6</sub>. No weed control (Control)

The different weeding methods were applied to control *Pennisetum polystachion* according to the schedule. As the chemical weeding (T<sub>1</sub>), glyphosate was applied at the rate of 1.44 ai kg/ha which is the recommended application rate of N-(phosphonomethyl)-glycine at 6 monthly intervals, during the latter part of the rainy season using a knapsack sprayer in the morning. Generally, there was no rain for five to six hours after applying glyphosate. The cover crop was established to control weeds and the over grown conditions of cover crop were managed to overcome competition by harrowing once a year. Tractor harrowing, slashing and ploughing were done at the latter part of the rain season at six monthly intervals.

#### **The data collected was as follows**

##### ***Pennisetum polystachion* biomass**

The weed biomass within 0.5m x 0.5m quadrates was collected from four random points from a plot. Weed samples were dried at 80°C for five days until they reached to a constant weight and the dry weight was recorded. The dry weight of weeds biomass was measured separately once in every two months from August 2014.

##### **Emergence of *Pennisetum polystachion* seedlings in the field**

In this study, four permanent quadrates (0.5m x 0.5m) were fixed randomly in each plot to monitor emergence of *Pennisetum polystachion* seedlings. The emerging seedling count was taken before and after imposing all treatments. The weeds which emerged around a 30cm border area outside each quadrate were removed frequently while the remaining area had free weed growth. The emerged seedlings within each quadrate were identified, counted and removed weekly for 12 weeks after applying the treatments. The average seedling density was estimated by summing the seedling count over the experimental period from August 2014 to May 2016.

##### **The seed depth of emerging *Pennisetum polystachion* seedlings in the field**

This study was done in 2014 Maha rain season (September – October), 2015 Yala rain season (April – May), 2015 Maha rain season (September – October) and 2016 Yala rain season (May – June). During the rainy season, the germination and emergence of *Pennisetum polystachion* seeds at different depths in the soil were measured in the field. Thirty (30) plants were used for this study. The weed seedlings were marked at ground level with Indian ink and each seedling was excavated to the depth of its caryopsis and the length from the caryopsis to the ink mark of each seedling was measured as described by Witharama (1998). The average seedling emergence depth was calculated by measuring the seedling emergence depth over the four rain seasons.

##### **The effect of treatments on tillering of *Pennisetum polystachion* rhizomes**

This study was carried out in 2014 Maha rain season (September – October), 2015 Yala rain season (April – May), 2015 Maha rain season (September – October) and 2016 Yala rain season (May – June). During the rainy season, the germination and emergence of *Pennisetum polystachion* rhizomes at different depths in the soil were measured in the field. Hundred (100) plants were used for this study.

Tillers were marked at ground level with Indian ink and each tiller was excavated to the depth of its rhizome and the length from the rhizome to the ink mark of each tiller was measured as described by Witharama (1998). The average emergence depth was calculated by measuring the tiller emergence depth over the four rain seasons.

**Data analysis:** Data were analysed statistically by the Procedure of Analysis of Variance (ANOVA) and means were separated by the Least Significant Difference test at the 0.05 significance level using Statistical Analysis System (SAS, 1999).

## Results and Discussion

### Effect of different weed control treatments on weed biomass

The lowest *Pennisetum polystachion* biomass was recorded in T<sub>1</sub> where glyphosate was applied at a concentration of 1.44 kg a.i. /ha and in T<sub>2</sub> where the *Pueraria phaseoloides* was established as a cover crop (Figure 1). With the application of glyphosate, the weed biomass has been reduced and weed seeds in the soil seed bank have been initiated their germination with the onset of rainy season. Most of the newly emerging weeds were annual dicotyledonous species and the emergence of *Pennisetum polystachion* was comparatively low.

Initially *Pueraria phaseoloides* took several months to establish a good ground cover. The *Pennisetum polystachion* biomass was very high at the initial stages in cover cropped plots which gradually declined later. With time *Pueraria* regenerated with seeds and formed a good ground cover, suppressing weed populations (Figure 1).

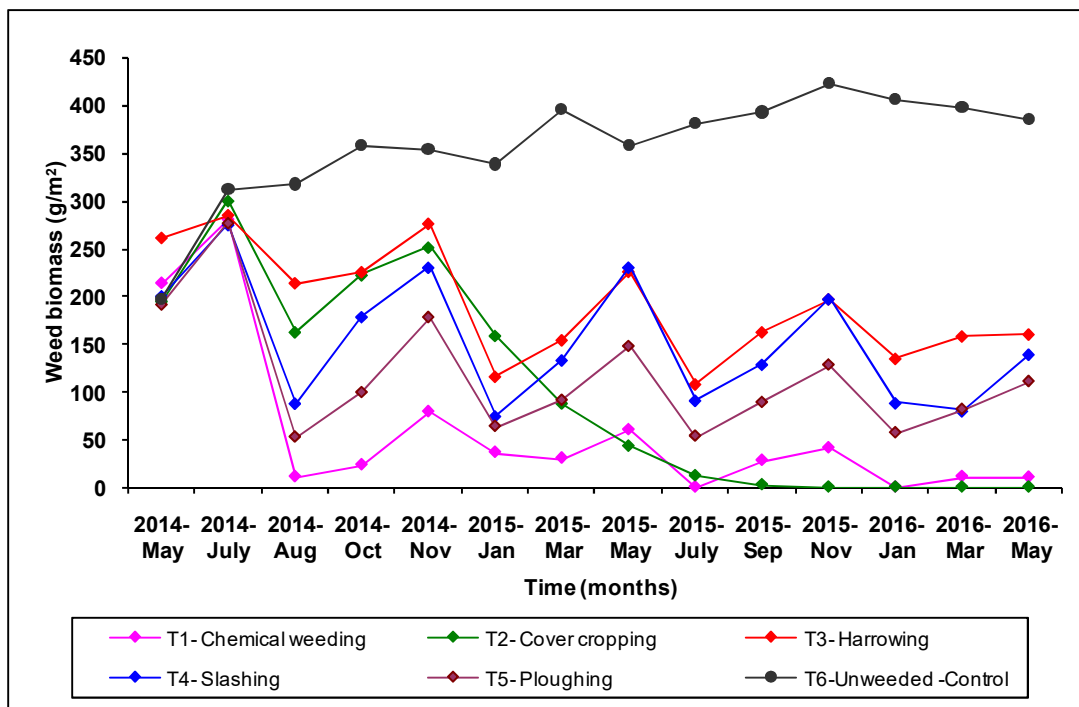
The three mechanical weeding treatments (tractor harrowing, tractor ploughing and tractor slashing) suppressed *Pennisetum polystachion* growth initially, but rapid re-growth was observed. Generally slashing damaged the aerial parts of the *Pennisetum polystachion* but no damage to the root system or underground plant parts. During favorable weather conditions, underground plant parts produced new shoots or

new flushes. However, slashing *Pennisetum polystachion* at shorter intervals in coconut lands may not be cost effective. Tractor harrowing and ploughing at six-month intervals reduced the *Pennisetum polystachion* biomass significantly compared to slashing. Both methods were helpful to bury weed seeds in deep layers, thus reduced the growth of weed population on the surface. However, this practice loosens the soil and which would create a suitable environment for the germination of some weed species seeds (Senarathne *et al.*, 2003).

### Weed seedlings emergence density in the field

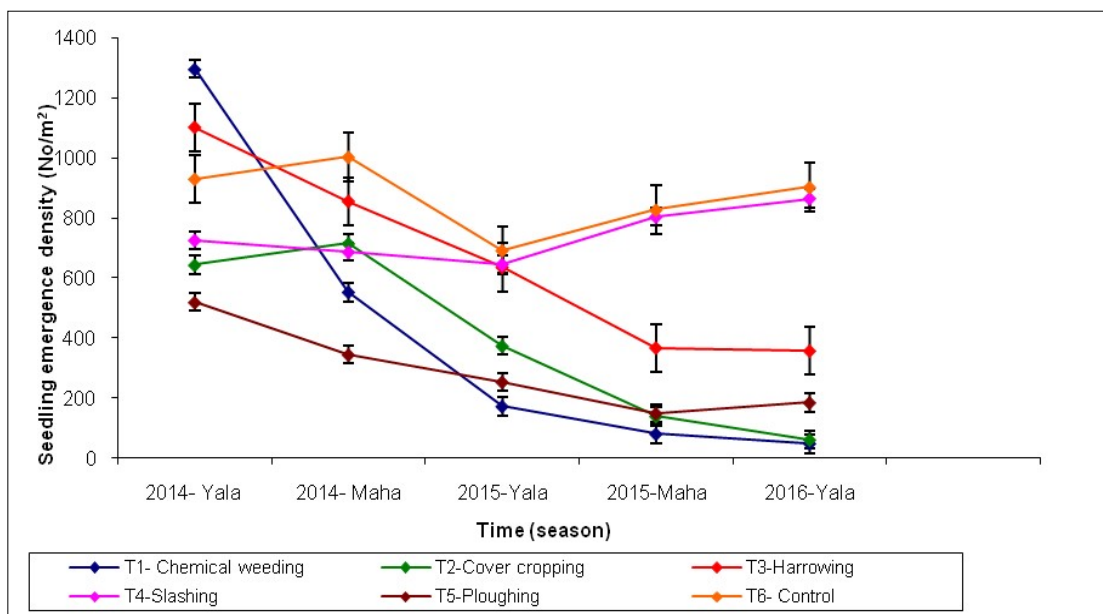
The numbers of *P. polystachion* weed seedlings gradually decreased with time in all weeding treatments except in the control plots (Figure 2). A high *P. Polystachion* weed seedling density was observed on the surface in the control and slashing treatments.

The *P. Polystachion* seedling emergence density was significantly low ( $P < 0.05$ ) in chemical and cover cropping treatments. The densities of emerged seedlings were almost similar in the chemically treated and cover cropped plots. The use of herbicides can also influence the species composition of the seed bank, seedling emergence density and may increase or decrease it, depending on the chemicals used (Ball, 1992), and can also cause specific shifts in weed populations (Roberts, 1968). The effectiveness of slashing is low in controlling *P. Polystachion* than cover cropping and chemical weeding methods and the weed seedling emergence density was comparatively lower in ploughed plots (Figure 2) than in the slashed and harrowed plots. Some weed species invaded a higher intensity of emergence in the no tillage planting than in the conventional tillage. The presence of seeds at the superficial layer of the soil and frequent cultivation, are factors that reduce the seed bank rapidly. This situation can facilitate seed loss by exposing seeds to variations in temperature and humidity, and breaking dormancy and finally reducing the seedling density in the field (Simpson *et al.*, 1989).



Treatments were applied in July 2014, November 2014, May 2015 and December 2015

**Figure 1. Effect of different weed management systems on total weed biomass from May 2014 to May 2016**



Treatments were applied in July 2014, November 2014, May 2015 and December 2015

**Figure 2. Effect of different weed management systems on *Pennisetum polystachion* seedling emergence density from 2014 to 2016**

**Table 1. Number of seedlings of *Pennisetum polystachion* emerged at different soil depths in different weed management treatments**

Treatments	Depths of emergence (cm)				Means emerged depth (mm)
	0-2cm	2-4cm	4-6cm	6-8 cm	
T <sub>1</sub> . Chemical weeding	18	8	4	0	07.58 <sup>c</sup>
T <sub>2</sub> . Cover cropping	22	6	2	0	05.55 <sup>d</sup>
T <sub>3</sub> . Harrowing	15	10	5	0	17.23 <sup>a</sup>
T <sub>4</sub> . Slashing	21	8	1	0	05.11 <sup>ed</sup>
T <sub>5</sub> . Ploughing	10	18	2	0	13.34 <sup>b</sup>
T <sub>6</sub> . Control	24	4	2	0	03.62 <sup>e</sup>
<b>Total</b>	<b>110 (61%)</b>	<b>54 (30%)</b>	<b>16 (8%)</b>		

- Values followed by the same letters are not different at P<0.05 in each treatment

**Table 2. Number of *Pennisetum polystachion* rhizomes germinated and emerged at different soil depths in different weed management treatments**

Treatments	Depths of emergence (cm)						Means emerged depth (cm)
	0 - 5cm	5- 10cm	10- 15cm	15- 20cm	20- 25cm	25- 30cm	
T <sub>1</sub> .Chemical weeding	12	5	0	0	0	0	3.2b
T <sub>2</sub> . Cover cropping	94	6	0	0	0	0	3.6b
T <sub>3</sub> . Harrowing	8	24	36	12	10	0	15.8a
T <sub>4</sub> . Slashing	93	7	0	0	0	0	3.4b
T <sub>5</sub> . Ploughing	11	18	38	26	7	0	18.3a
T <sub>6</sub> . Control	95	5	0	0	0	0	2.8b

- Values followed by the same letters are not different at P<0.05 in each treatment

In this study, chemical weeding and cover cropping were the best methods to reduce weed seedling emergence density. This may depend on several factors, including the pattern of rain fall and the time of germination at a site, the timing of seed input (seed rain) into the seed bank and different agronomic practices (Coffin and Lavenroth, 1989) and seed and seed losses due to predators (Hodgkinson *et al.*, 1980; Rice, 1989). However, different weeding methods over the experimental period in different treatment plots produced dense stands of weeds and the seed rain from these plants probably caused the seed bank changes observed in subsequent sampling occasions.

#### **The seed depth of emerged *Pennisetum polystachion* seedlings in the field**

The average depths of *P. Polystachion* seedlings in the field are presented in Table 1. The seed depth of emerged *P. Polystachion* seedlings was higher in harrowed and ploughed weeding treatments compared to other weeding treatments. The large number of seedlings of *P. polystachion* (61%) in chemical weeding, cover cropping, slashing and control treatments were derived from the seeds in the top 1cm of the soil. In harrowing and ploughing plots, *P. Polystachion* seedlings emerged from depths of 1.7cm and 1.3cm of the soil.

However, the optimum depths for the emergence of seeds vary with different species. By compiling data for 31 species, King (1966) showed that optimum depth ranged from 0.05cm to 2.5cm and was roughly in proportion to the 1000 seed weight. Roberts and Feast (1973) examined the depth of seed burial in undisturbed and cultivated soil and found seedling emergence was greatest from cultivated soil at shallow depths of burial.

#### **The effect of different treatments on tillering of *Pennisetum polystachion* rhizomes**

Table 2 shows the effect of different ground cover management systems on the tillering of *Pennisetum polystachion* rhizomes. The highest mean emerged depth was recorded by the ploughing treatment (T<sub>5</sub>) and the next highest mean emerged depth was given by the

harrowing treatment (T<sub>3</sub>). Results given by T<sub>3</sub> and T<sub>5</sub> indicates that turning soil enables the rhizomes from deeper soil layers to produce the new tillers. However, the deeper the depth of the soil, lesser the number of emergence of tillers.

This indicates that loosening the soil, creates more favourable environment for the rhizomes in deeper layers to produce tillers.

#### **Conclusion**

Application of glyphosate (1.44 kg a.i./ha) and cover cropping (*Pueraria phaseoloides*) methods are more effective to reduce *Pennisetum polystachion* biomass and seedling emergence density compared to other weeding methods such as harrowing, slashing and ploughing. Considering the soil seed bank, the results of this study have provided useful information on timing, emergence density and composition of weed populations that are likely to emerge under different types of weed management systems in relation to the seed bank. However, the depths of weed seedling emergence were very high in harrowing and ploughing treatment plots when compared to the other weeding methods. This indicated that loosening the soil creates more favorable environment for germinate weed seeds buried in deep soil layers.

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**The introduction** should have a brief statement of the problem and explain the aim or the objectives of the investigation. **The materials and methods** should be very clear with all details of experimental design, treatments, location, period of study, methods adopted etc. Methods should be clearly written so that the reader of your article should be able to use it in pursuing her/his studies elsewhere.

**The results and discussions** should provide data organized into tables, figures and photographs, suitably compared and discussed with earlier published findings. All data must be presented in metric units.

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