A Case Study of RIT-Pilot Plant for Thai Neem-based Extract Processing : From Research in BRD to Small-scale Industrial Production in Thailand [1]

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Abstract

Neem-based extract processing at Rajamangala Institute of Technology (RIT) in Patumtani province involves a long chain of operations and various equipment. The steps are : seed decorticating, crushing, oil expel, agitation, filtration, evaporation and formulation. As raw material, Thai neem seeds (*Azadirachta indica* var. *siamensis* (Valeton)) are used. They are firstly decorticated to obtain the seed kernel, then crushed and finally pressed to separate neem oil by screw expellor. By moving-bed contacting extraction technique, defatted neem cake will be extracted with methanol in an agitated-extraction vessel. After decantation of crude cake in mixing-settling tank, the neem solution is drained out, then filtered and proceeded to the next procedure. The solution will be further evaporated until a specific volume, the so-called-concentrated alcoholic neem-based extract. Before packing in containers, the concentrate will be analyzed for azadirachtin ($C_{36}H_{44}O_{16}$) content by using HPLC. Furthermore the concentrate is also formulated for specific purpose as different commercial grade. Eventually, the product will be bottled and shipped to the consumer.

The described processing requires a set of special equipment, such as seed decorticator, pulverizer, oil expeller, filter, agitated-extraction vessel and evaporator. However, the actual yield of different neem-based extracts by all equipment and processes was compared with yield of laboratory scale, which was used as standard method or control treatment. The data obtained are discussed in terms of development and improvement for further manufacturing.

KEYWORDS:
neem-based extract; moving-bed contacting; agitated-extraction vessel; vacuum evaporator; *Azadirachta indica* var. *siamensis*

1. Introduction

The efficacy of neem-based extracts displays an array of effects on insects, such as repellent, antifeedant, growth-retardant, molt disrupting, progeny development disrupting and oviposition deterrent. (National Research Council, 1992; Schmutterer, 1995). Although every plant part of the neem tree contains pest control properties, the past research works indicated that active ingredients are mostly concentrated in the seeds. Azadirachtin ($C_{36}H_{44}O_{16}$), tetranortriterpenoid, the most active insecticidal substance in neem seed, causes growth disruption, molting inhibition, egg-sterilization and other effects (Schmutterer, 1995). In Thailand, neem could be of economic significance and practically applied in many rural areas. It shows considerable potential for controlling of various insect pests such as *Plutella xylostella, Spodoptera litura, S. exigua, Hellula undalis, Phyllocnistis citrella, Helicoverpa armigera, Ohiomyia phaseloi, Nephotettix virescens* (Sombatsiri *et al.* 1995) and phytophagous mites (Sanguanpong and Schmutterer, 1992) . Besides, the practice of mixing neem materials especially neem oil with store products in a warehouse trial showed an effective protection against certain store insect pests (Sanguanpong *et al.*, 2001a, 2001b, 2002 a**) .**

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Sadao, the local name of Thai neem, *Azadirachta indica* var. *siamensis* (Valeton), can be found in the North, Central, Northeast and South of Thailand. It is popularly used as farm borders and roadsides-tree and grows well at altitudes below 200 meters. Similar to "Sadao-Thai", the other two varieties, Indian neem tree (*A. indica* A. Juss) and Marrango tree (*A. excelsa* (Jack) Jacobs), are naturally found in western and southern part of Thailand, respectively. According to their natural widespread, Thai neem is considered as the main kind of raw material for a commercial production in Thailand.

A single-step extraction method is commonly used for commercial production of neem-based extract in Thailand. Figure 1 shows the schematic diagram of of Thai neem-based extract processing in small-sized manufactures(Sanguanpong, 2000). Neem seeds are firstly crushed to crude powder and then extracted with methanol. By moving-bed contacting method neem seed will be stirred for 3-4 hours by overhead stirrer in mixing-settling tank. After decantation of crude cake, the neem solution is drained out, then filtered and proceed to the next procedure. The dilute neem solution will be further evaporated until a specific volume-the so called-"concentrated extract". Eventually, the obtained solution can be bottled and shipped to the consumer. Furthermore some of the products are formulated for a specific purpose. However, the formulation technology of neem-based insecticides is commercial secret.

According to the single-step extraction method, the concentrated extract still contains neem oil, which actually causes phytotoxic to the plant, if the oil content exceeds 1.00 % W/W (Srivastava and Parmar ,1985). In some cases "latent phytotoxicity" by higher concentration of neem-based extract can result in lower yields than do lower doses (Ermel and Kleeberg, 1995). To separate the oil from neem material, oil expellor was designed and constructed. Besides, in this current experiment various equipment for other operations were also designed and assembled at Faculty of Agricultural Engineering and Technology as prototype for small-scale industrial production, such as seed decorticator, pulverizer, agitated-extraction vessel and evaporator. The actual yield of different neem-based extracts by all equipment and processes was compared with data on productivity under processing in laboratory, which was used as standard method or control treatment. The data obtained are discussed in terms of development and improvement for further manufacturing.

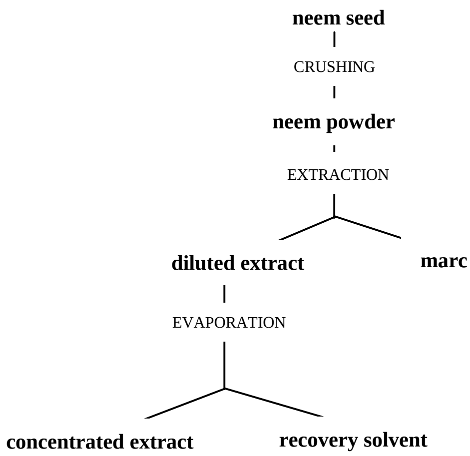

Figure 1 Schematic diagram of of neem-based extract processing in small-sized manufactures in Thailand

2. Materials and Methods

2.1 Processing Technology and Machinery

As raw material for neem-based extract processing, Thai neem seeds (*Azadirachta indica* var. *siamensis*) are used. Figure 2 shows the schematic diagram of Thai neem-based extract processing at RIT-Pilot Plant resulting with equipment. Thirty kilogram of dried neem seeds at 8-12 %(w.b.) moisture content was used in each experiment. They are firstly decorticated to obtain the seed kernel, then crushed and finally pressed to separate neem oil. By moving-bed contacting extraction technique, defatted neem cake will be extracted with methanol (1:3 W/W) in an agitated-extraction vessel. After decantation of crude cake in mixing-settling tank, the neem solution is drained out, then filtered and evaporated until a specific volume, the so-called-concentrated alcoholic neem-based extract. After quality measurement, the concentrate could be formulated for specific purpose as different commercial grade. Eventually, the product will be bottled and shipped to the consumer.

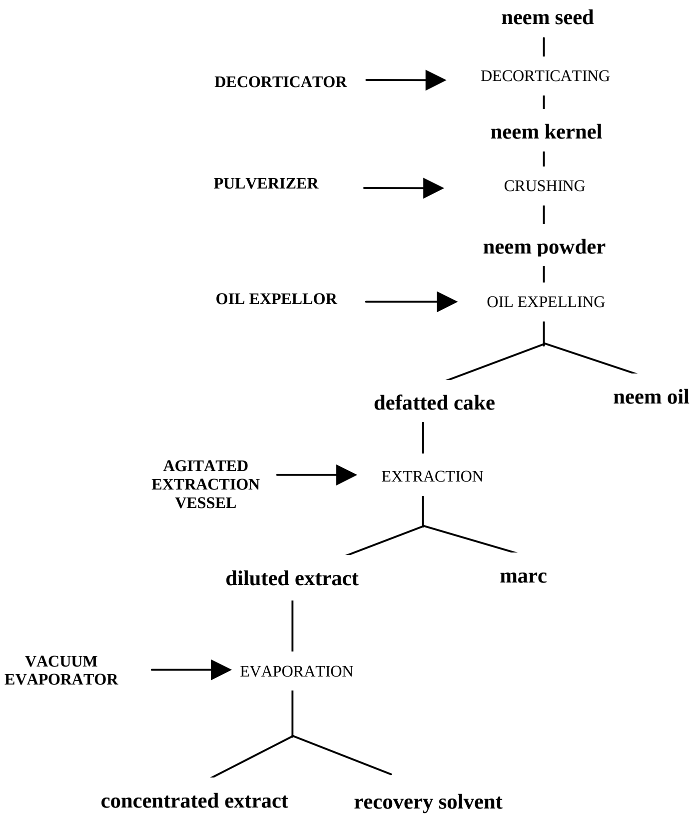

Figure 2 Schematic diagram of neem-based extract processing in RIT-Pilot plant resulting with equipment

2.2 Quality Control by Chemical Analysis of Azadirachtin

According to registration of neem-based extract for domestic production and use in Thailand, the requirements at the Toxicity Substance Act B.E. 2510 (1967) and B.E. 2516 (1973) under chemistry are prescribed by not requiring the toxicological data (Wong-Ek *et al.*, 1997). The registration guideline set by Department of Agriculture, requires that a neem formulation should contain at least 0.1% azadirachtin (Praneetwattakul *et al.*, 1999). Hence, before packing and labelling, the concentrate will be analyzed for azadirachtin ($C_{36}H_{44}O_{16}$) content. It has been characterized quantitatively mainly by using High Performance Liquid Chromatography (HPLC) as described by Schneider and Ermel (1987).

2.3 Process Monitoring

To monitor the performance of two main processes, extraction and evaporation, the experiment under the same procedure as in pilot plant will be carried out in laboratory conditions and used as control process. 30 g of defatted neem cake was extracted with methanol (1:3 W/W) by moving-bed contacting using 8 positions magnetic stirrer. After decantation of crude, the solution is drained out, then filtered and further evaporated by rotary vacuum evaporator. The concentrate neem-based extract was residued after complete removal of methanol.

2.4 Characteristics of Physical Properties

Even information on the physical properties such as L-a-b value (Hunter system), total soluble solid (TSS, °Brix) and density of neem-based extract as compared with chemical analysis of azadirachtin is yet not practical. It could be proper for rapid comparative analysis of large number of commercially available products and for optimizing process control. The L-a-b values of the extract were measured by Tristimulas-Colorimeter (Juki JC-801S, Japan), while refractometer was employed to measure total soluble solid (TSS, °Brix). Density was also measured in terms of mass (kg) per volume (liter) in all samples.

3. Results

3.1 Processing Technology and Machinery

Data on preconditioning of neem materials by decorticating, crushing and oil expel was given in Table 1. By decorticating of Thai neem seed, the constitution of 50.17 ± 3.08 % hull and 47.75 ± 3.05 % kernel were found, while processing loss of 2.08 ± 0.44 was obtained. The same trend on decorticating of Indian neem seed using 2 different disc hullers, i.e. granite disc huller and emery disc huller was reported by Ramakrishna *et al*. (1996). It was noticed that indian neem seed constituted of 53 % hull and 35 **%** kernel, and the emery disc huller was most efficient in decorticating. However, it was not mentioned on moisture content of seed materials, which can influence on result data (Sivakumar *et al*., 1996). In the next processing, seed kernel was directly fed into pulverizer (hammer mill). Data obtained showed that crushing of seed kernel gave also a good yield. However, processing loss of 6.67 ± 3.33 were observed in crushing. Through oil expellor, only 7.93 ± 1.79 % neem oil was obtained, while the oil content is 18.64 ± 2.28 % in whole seed. It is suggested that development of oil expellor should be considered for better manufacturing.

Table 1 Preconditioning of neem material (8-12 % mc) by decortication, crushing and oil Expelling

Process (Equipment)	Products	Yield (%)
Decorticating (Decorticator)	Hull*	50.17 ± 3.08
	Kernel	47.75 ± 3.05
	Processing loss	2.08 ± 0.44
Crushing (Pulverizer)	Kernel powder	93.33 ± 3.33
	Processing loss	6.67 ± 3.33
Oil expelling (Oil expeller)	Oil**	7.93 ± 1.79
	Defatted cake	88.10 ± 2.69
	Processing loss	3.97 ± 0.90

* Thai neem seed constitutes of 50.07± 1.07% hull and 49.93±1.12 % kernel
** The oil content is 18.64 ± 2.28 % in whole seed.

Data on productivity of extraction and evaporation of neem-based extract was given in Table 2. It was found that extraction process gave a good yield of solution and a few loss of solvent was also determined. Compare to evaporation process, loss of solvent at 13.72 ± 0.34 % was observed. This reflected the low performance of evaporator which could be considered for further improvement. Similar data was reported on vacuum evaporator for village scale (capacity of 30 L/batch), but fewer loss of solvent (8.00 ± 3.60 %) was determined (Sanguanpong, 2002 b).

Table 2 Productivity of neem-based extract under processing in RIT-pilot plant

Process	Solution out [1] (%)	Marc out [2] (%)	Recovery solvent [3] (%)	Loss of solvent [4] (%)
Extraction	63.67 ± 3.79	27.00 ± 4.00	-	9.33 ± 1.53
Evaporation	50.17 ± 1.09	-	36.11 ± 1.17	13.72 ± 0.34

[1] solution out (%) = (weight of extract)* 100 / total mass

[2] marc out (%) = (weight of marc)* 100 / total mass

[3] recovery solvent (%) = (weight of recovery solvent)* 100 / total mass

[4] loss of solvent (%) = (total mass – solution out -marc out) * 100 / total mass

3.2 Quality Control by Chemical Analysis of Azadirachtin

The result of azadirachtin and oil content was shown in Table 3. Only 0.98 ± 0.06 % oil was found in the concentrated extract, whereas the average amount of azadirachtin content of Thai neem-based extract was 3.43 ± 0.64 mg/ g seed kernel. Compare to Foerster and Moser (2000), it was reported that azadirachtin content of neem kernel in Thailand was approx. 5.20 mg/ g, whereas in India only 5.14 mg/ g was found. High amount of azadirachtin were observed in Ghana and Kenya about 6.2-6.9 and 6.8-8.8 mg/ respectively. However, many important factors can affect the degradation of azadirachtin in tropical regions, i.e. high temperature, humidity and storage conditions (Ermel *et al.*, 1987). Hence, it is difficult to define the ability or quality of process by using omly azadirachtin content in material. These could be confirmed by Sombatsiri *et al.* (1995), who found that samples from different provinces throughout Thailand contained azadirachtin content from inferior quality (1.40 mg/g) to high quality (5.30 mg/g).

Table 3 Azadirachtin (amount in mg/ g and concentration in solution) and oil content (%) in neem-based extract produced from RIT Pilot Plant

Production Scale	Azadirachtin content		Neem oil (%)
	mg/ g ± SE	% in solution	
Pilot Plant	3.43 ± 0.64	0.23 ± 0.02	0.98 ± 0.06

3.3 Process Monitoring

In Table 4 data obtained on productivity of extraction and evaporation of neem-based extract under laboratory scale are presented. Compare to productivity under processing in RIT-pilot plant in Table 2, the results confirmed that the extraction process in RIT-pilot plant gave a good yield as same as in laboratory but more loss of solvent was determined. Similar to extraction process, more loss of solvent was observed by evaporation in RIT-pilot plant. Yield and loss (%)

of different products under the same process in different scales were also calculated (Table 5). It showed that yields obtained from pilot plant were not different from control process. On the other hand, loss of solvent by extraction and evaporation process in pilot plant were relative higher than in laboratory.

Table 4 Productivity of neem-based extract under processing in laboratory

Process	Solution out [1] (%)	Marc out [2] (%)	Recovery solvent [3] (%)	Loss of solvent [4] (%)
Extraction	68.49 ± 1.33	30.42 ± 1.26	-	1.09 ± 0.16
Evaporation	50.01 ± 0.01	-	46.40 ± 1.25	3.60 ± 1.25

[1] solution out (%) = (weight of extract)* 100 / total mass
[2] marc out (%) = (weight of marc)* 100 / total mass
[3] recovery solvent (%) = (weight of recovery solvent)* 100 / total mass
[4] loss of solvent (%) = (total mass – solution out -marc out) * 100 / total mass

Table 5 Yield and loss (%) of different products under processing in laboratory compare to RIT- pilot Plant

Production scale	Neem-based extract [2] (%)	Marc out [2] (%)	Recovery solvent [3] (%)	Loss of solvent [4] (%)	
				Extraction	Evaporation
Laboratory	34.25 ± 0.01	30.42 ± 1.26	31.77 ± 1.25	1.09 ± 0.16	2.47 ± 1.25
Pilot plant	31.57 ± 2.71	27.00 ± 4.00	22.67 ± 0.76	9.33 ± 1.53	8.63 ± 0.71

[1] solution out (%) = (weight of extract)* 100 / total mass
[2] marc out (%) = (weight of marc)* 100 / total mass
[3] recovery solvent (%) = (weight of recovery solvent)* 100 / total mass
[4] loss of solvent (%) = (total mass – solution out -marc out) * 100 / total mass

3.3.2 Characteristics of Physical Properties

Characteristic comparison on physical properties of neem-based extract produced from laboratory and RIT-pilot plant was shown in Table 6. Under the same procedure, neem-based extract could be produced more concentrate in RIT-pilot plant than those obtained in laboratory. More total soluble solid (°Brix) and density were observed. L-value(Lightness value) indicated that the extract from laboratory was brighter than from pilot plant. It was obviously seen from the color of neem-based extract from pilot plant, which was yellow-brown and darker than from laboratory. However, the relevance between physical properties and quality of neem product was not investigated.

4. Conclusions

Small-scale industrial production of neem-based extract in RIT- pilot plant was studied. A set of special equipment, such as seed decorticator, pulverizer, oil expeller, agitated-extraction vessel and evaporator were assembled. They were employed to produce mainly an alcoholic neem-based extract. The performance of all equipment was evaluated. With 250 days running, the capacity of pilot plant will be approx. 27,000 L/year. However, low capacity of some equipment, in particular, oil expellor and high processing loss of evaporator were found. To apply the finding for improvement of processing quality, it is still necessary to develop a suitable equipment for further manufacturing.

Table 6 Physical Properties of neem-based extract produced from RIT-pilot plant compared to from laboratory

Production Scale	L-a-b value			° Brix	Density (Kg/L)
	L	a	b		
Laboratory	43.57 ± 0.71	-1.89 ± 0.80	29.25 ± 0.35	8.00 ± 0.00	0.82 ± 0.00
Pilot Plant	28.86 ± 6.44	1.13 ± 1.42	19.97 ± 4.57	10.33 ± 2.89	0.83 ± 0.01

On these attributes, data analysis of productivity in laboratory was recommended for process monitoring and optimizing. However, the ability to optimize or improve the process is dependent upon not only the ability to control the process, but also the access to reliable eventually valid measurements. By the way, requisite research on physical properties of neem-based products and development effort will have to put not only technology practically but also technological challenge.

Due to increasing of consumer health awareness in case of toxic residues in food crops, it can be expected that market potential of neem-based pesticide will be growth. At present, three kinds of neem-based products are availble : RITNEEM (a liquid extract containing 0.10-0.30 % aza.), RITNEEM -DC (defatted neem cake for "Tea-bag method") and RITNEEM –O (formulated neem oil). However, the products are not available in the market. They are only produced for distribution to RIT-agricultural campus in different part of Thailand. Through multilateral co-operation with representatives of companies and some institution (i.e. Federal of Thai Industrial), chance for joint-venture are being done.

5. Acknowledgement

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