

PRODUCTIVITY OF RIT-PILOT PLANT FOR SMALL-SCALE INDUSTRIAL PRODUCTION OF THAI NEEM-BASED EXTRACT

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ABSTRACT

A pilot plant of 25 Ton/year for the manufacturing of ready-to-use neem-based pesticide was setting up since 2001. The process involves a long chain of operations and requires a set of special equipment, such as seed decorticator, pulverizer, oil expeller, filter, agitated-extraction vessel and evaporator. 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 moving-bed contacting technique, defatted neem cake will be extracted with methanol. After decantation of crude cake, 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 neem-based extract. Before packing, the concentrate will be analyzed for azadirachtin (C₃₆H₄₄O₁₆) content by using HPLC. Furthermore it is also formulated for specific purpose as different commercial grade. Eventually, the product will be bottled and shipped to the consumer. However, the actual yield of different neem-based extracts in each process was compared to yield of laboratory scale, as standard method. The data obtained are discussed in terms of development and improvement for further manufacturing.

KEYWORDS:

neem-based extract, *Azadirachta indica* var. *siamensis*, seed decorticator, agitated-extraction vessel, evaporator

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 (C₃₆H₄₄O₁₆), 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 phaseloii*, *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).

Sadao, the local name of Thai neem, *Azadirachta siamensis* syn. *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 Kleeborg, 1995). To separate the oil from neem material, oil expeller 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.

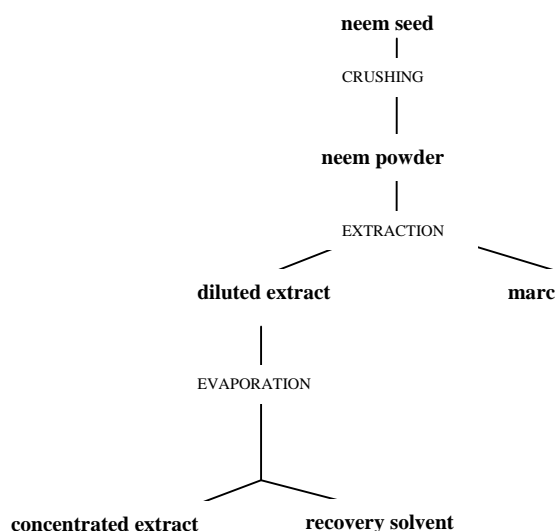


Figure 1 Schematic diagram of Thai neem-based extract processing in small-sized manufactures in Thailand

2. Materials and Methods

2.1 Processing Technology and Machineries

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 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.

2.2 Quality Control

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).

Even the information on 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

Tristimulus-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.

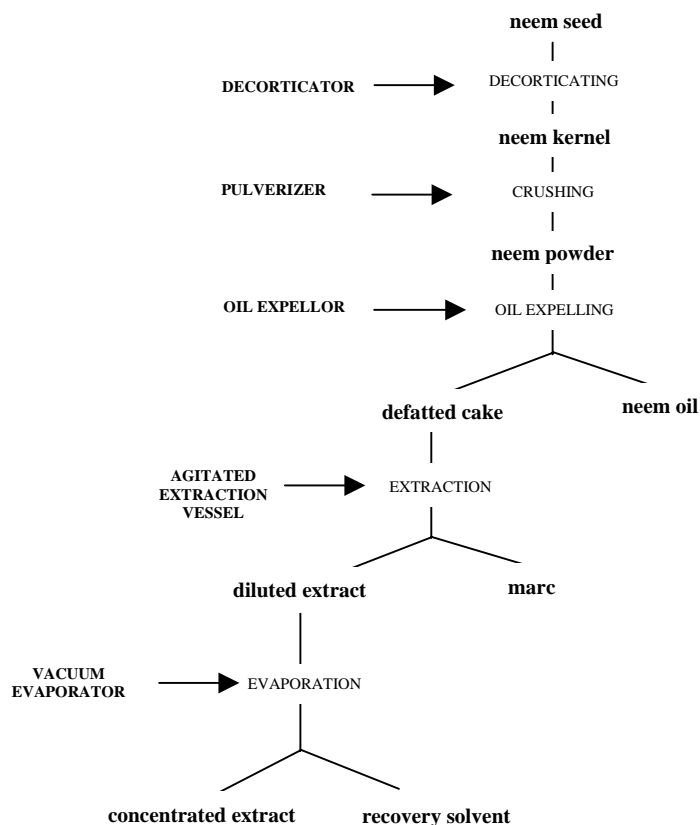


Figure 2 Schematic diagram of neem-based extract processing in RIT-Pilot plant resulting with equipment

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. Thirty grams 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. Data on productivity of extraction and evaporation of neem-based extract was measured and compared to pilot plant.

3. Results

3.1 Processing Technology and Machinery

Data on preconditioning of neem materials by decortivating, crushing and oil expel was given in Table 1. By decortivating 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 decortivating 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 decortivating. 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 expeller, 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 expeller should be considered for better manufacturing.

Table 1 Pre-conditioning of neem material (8-12 % MC) by decorticating, crushing and oil expel

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

* In laboratory trial 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).

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

Type of products	Process	
	Extraction	Evaporation
Solution out	63.67 ± 3.79	50.17 ± 1.09
Marc out	27.00 ± 4.00	-
Recovery solvent	-	36.11 ± 1.17
Loss of solvent	9.33 ± 1.53	13.72 ± 0.34

3.2 Quality Control

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 $3.43 \pm$

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 (Ernel *et al.*, 1987). Hence, it is difficult to define the ability or quality of process by using only 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 Comparison on chemical and physical properties of neem-based extract produced from laboratory and RIT-pilot plant

Observed data	Production scale	
	Pilot plant	Laboratory
Azadirachtin content ¹	3.43 ± 0.64	3.43 ± 0.64
Azadirachtin content ²	0.23 ± 0.02	0.23 ± 0.02
Neem oil ²	0.98 ± 0.06	0.29 ± 0.00
L-value	28.86 ± 6.44	43.57 ± 0.71
a-value	1.13 ± 1.42	-1.89 ± 0.80
b-value	19.97 ± 4.57	29.25 ± 0.35
° Brix	10.33 ± 2.89	8.00 ± 0.00
Density (kg/L)	0.83 ± 0.01	0.82 ± 0.00

¹ Azadirachtin content in mg/g \pm SE

² Azadirachtin and oil content in (%)

Characteristic comparison on physical properties of neem-based extract produced from laboratory and RIT-pilot plant was also shown in the last given Table. 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 is being investigated.

3.3 Process Monitoring

Yield and loss (%) of different products under the same process in different production scales were shown in Table 4. The results confirmed that yields obtained from pilot plant were not different from control process. The extraction in RIT-pilot plant gave a good yield of neem-based extract and marc out as same as in laboratory. On the other hand, loss of solvent by extraction and evaporation process in pilot plant were relative higher than in laboratory.

Table 4 Yield and loss (%) of different products under processing in laboratory compare to RIT- pilot Plant

Type of materials	Yield and loss (%) under different production scale	
	Pilot plant	Laboratory
Neem-based extract	31.57 ± 2.71	34.25 ± 0.01
Marc out	27.00 ± 4.00	30.42 ± 1.26
Recovery solvent	22.67 ± 0.76	31.77 ± 1.25
Solvent loss in extraction	9.33 ± 1.53	1.09 ± 0.16
Solvent loss in evaporation	8.63 ± 0.71	2.47 ± 1.25

4. Discussion

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. 25,000 L/year. However, low capacity of some equipment, in particular, oil expeller 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.

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 available: 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, small or medium entrepreneurs are being considered as target group for transferring the technology.

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