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SCALE-UP OF EXTRACTION PROCESS FOR DILL SEED OIL

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Keywords:

Dill seed oil, Extracts, Energy Balance, Raw materials, Scaling Up, Soxhlet system

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ABSTRACT: Concentrated preparations called extracts are made from raw materials by isolating specific components through specific methods. Essential oils, tinctures, liquid extracts, and dry extracts are among the different kinds of extracts. The type of input material, the solvent employed, the temperature, and the extraction time are all important variables. Efficiently isolating and condensing particular ingredients for use in tastes, perfumes, and medicinal applications is the aim of extraction. A Soxhlet system can be used to manufacture dill seed oil through continuous solvent extraction. The right solvent must be used for this procedure; ideally, it should be able to dissolve the targeted compounds without causing any degradation. In order to extract the oil, the Soxhlet extraction cycle involves heating the solvent, letting it evaporate and condense, and then repeatedly washing the raw material. Requirements for scaling up Soxhlet extraction include preserving steady-state mass and energy balances. While the energy balance takes into consideration the heat input and thermal losses during the process, the mass balance makes sure that the amounts of the input raw materials and solvent match those of the extracted product and residues. Comprehending these equilibriums is essential for maximizing effectiveness and guaranteeing repeatability in larger-scale procedures. In conclusion, in order to produce high-quality extracts fit for commercial use, efficient extraction using the Soxhlet apparatus necessitates careful condition selection and a deep comprehension of both mass and energy balances.

INTRODUCTION:

Describe Extraction Process: The objective of the extraction process is to remove the insoluble fraction of the residue and separate the soluble plant metabolites. The end product, referred to as the extract, is a rather complex blend of metabolites that might be liquid, semisolid, or dried powder if water reduction is the goal. Three fundamental factors affect the extraction quality. The extraction process, the plant portion utilized as a source material, and the solvent used.

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The most commonly used extraction method for producing an extract is solvent extraction. The safety, cost, selectivity, and solubility of a solvent should all be considered. The effectiveness of solvents with polarity values near the polarity of the solute is probably higher. In accordance with the laws of similarity and intermiscibility, vice versa.

Alcohols (MeOH and EtOH) are the two most commonly utilized solvents in solvent extraction for phytochemical studies. Additional solvents used include hexane, acetone, ethyl acetate, petroleum ether, n-butanol, chloroform, benzene, water, and methylene chloride. Preservative action, low toxicity, inertness, resistance to easy combustion, minimal or no chemical interaction, facilitation of the extract’s quick physiological absorption, low heat evaporation ease, and the extract's incapacity to complex or dissociate are all considered characteristics of an exceptional extraction solvent.

Purpose of Extraction: When choosing an extraction method, factors including the required active compound properties, the plant material's water content, and the process goals are usually taken into account. Getting the targeted compounds with the greatest quality and largest yield is the main objective of the extraction procedure. The bioactivities of natural BCs are determined by *in-vitro* and *in-vivo* testing after they are extracted using a variety of extraction procedures.

The extraction of BCs is dependent on numerous factors, such as the extraction technique, raw materials, and solvent. The extraction of BCs is dependent on numerous factors, such as the extraction technique, solvent, extraction time, raw materials, temperature, solvent pH, pressure, tissue particle size, solvent-to-sample ratio, etc. The following are the main objectives of extraction techniques: Targeted BCs can be extracted from complex plant materials in four ways:

A. To enhance the concentration of the target compounds in bioassays to increase sensitivity.

B. To improve analytical method selectivity.

C. To transform the BCs into a format that is more appropriate for screening and separation.

D. To provide a dependable and repeatable technique that is unaffected by modifications in the sample matrix [2].

Extraction Process Types: There are two categories of extraction processes:

- Traditional or conventional **Table 1**.
- Inventive or nonconventional **Table 2.**

In addition to traditional methods, other innovative approaches have been developed, but no method is currently recognized as the gold standard for extracting BCs from plants. Since, different polarity solvents are needed for the identification and separation of the target component, the polarity of the chemical and the solvent choice have an impact on the efficacy of conventional extraction techniques. More mass and heat transfer, a faster extraction rate, more efficient energy consumption, smaller equipment dimensions, and fewer processing steps are the goals of these green extraction techniques. Traditional extraction techniques, such as hydro distillation and Soxhlet, are still regarded as a means of evaluating the efficacy of the recently established technology [2].

TABLE 1: CLASSIFICATION OF CONVENTIONAL OR TRADITIONAL TECHNIQUES FOR BIOACTIVE COMPONENT EXTRACTION [2]

System	Binomen	Effects	Plant-derived
Steam distillation	Aerieal parts */Rosmarinus officinalis.*	Antioxidant activity	Borneol acetate, eucalyptol, camphor, and β-caryophyllene.
Infusion	Aerial parts/*Salvia amarissima* Ortega.	Hypoglycemic effect	5,6,4′-trihydroxy-7,3′-,6-hydroxyluteolin, rosmarinic acid, and Amarisolide.
Mechanical pressing	*Cucurbita moschata*/oil and *Cucurbita argyrosperma.*	Antioxidant activity	Tocopherols: α-tocopherol, stigmasterol. Carotenoids: lutein, β-cryptoxanthin, and β-carotene.
Serial exhaustive extraction	Aerieal parts */Schkuhria pinnata* (Lam).	Anti-mycobacterial activities and antioxidants	Eucannabinolide and 6α-[4′,5′ dihydroxytigloyloxy]-isoinuviscolide, 6α [4′,5′dihydroxytigloyly]-inuviscolide, 3-desacetyl-3-isovaleroyleucannabinolide.
Agitation	*Hedeoma patens*/leaves, *Lippia graveolens*, and *Lippia palmeri.*	Anti-inflammatory potential	6-O-methylscutellarein, Quercetin O-hexoside, and scutellarein 7-O-hexoside.
Reflux extraction	Fruit*/Opuntia ficus-indica* mill.	Anti-helminthic activity, fungicidal, bactericidal, antioxidant, antiseptic	Isorhamnetin, Trans-caffeic acid, kaempferol, ferulic acid, and quercetin.
Maceration	Aerial parts*/Lippia graveolens.*	UV protection/Antioxidant	Pinocembrin, Naringenin, and galangin.
Fermentation	*Agave*	Anti-inflammatory	Glycosides, Saponins, and terpenoids.

(aqueous-alcoholic extraction)			
Hydrodistillation	*Leaves/P. longiflora.*	Anti-microbial activity and antioxidant activity	Thymol acetate, phenolic acids, Methylmaleic anhydride, thymol, carvacrol acetate, and thymoquinone.
Digestion	*Annona marcgravii*/fruit, *Carica papaya*, and *Artocarpus heterophyllus.*	Antioxidant activity	Carotenoids, chlorophyll α and chlorophyll β.
Lixiviation (elution)	*Fruit/Capsicum annuum.*	Antioxidant capacity	Carotenoids and Capsaicinoids.
Percolation	Leaves and flowers */L. graveolens.*	Anti-inflammatory, anti-glycemic and cytotoxic.	Cirsimaritin, 3-O-methylgalangin, Thymol, and Carvacrol, Cirsimaritin.
Decoction	Flowers/*Hibiscus sabdariffa* L.	Antioxidant activity	5-(hydroxymethyl) furfural, 3-O-caffeoylquinic acid, α-Tocopherol.
Hot continuous extraction or Soxhlet extraction	*Cucurbita moschata*/oil and *Cucurbita argyrosperma.*	Antioxidant activity	β-cryptoxanthin, Tocopherols: α tocopherol, Carotenoids: lutein, δ-tocopherol, γ tocopherol, β-sitosterol, stigmasterol.

TABLE 2: BIOACTIVE CHEMICAL EXTRACTION TECHNIQUES ARE CLASSIFIED ACCORDING TO THEIR NOVEL OR NONTRADITIONAL APPROACHES 2

System	Binomen	Effects	Plant-derived
Pressurized liquid extraction (PLE) or accelerated solvent extraction (ASE)	Peel and pulp/ *Opuntia ficus-indica* (L.) mill	Anti-inflammatory activities and Antioxidant	Ascorbic acid, phenolics and Betalains,
High-voltage-assisted extraction	Cocoa bean shell	Antioxidant activity	Gallic acid, Catechin and epicatechin
Turbo-distillation extraction or turbo-extraction (turbolysis)	Aerial parts/ *Cinnamomum cassia, Schinus terebinthifolius and Illicium verum*	-	α-pinene, and β-myrcene, α-phellandrene, limonene + β-phellandrene
Microwave-assisted extraction (MAE)	Aerial parts/*Ocimum basilicum* L.	Antioxidant	b-cubebene, linalool, Methyl cinnamate and eucalyptol
Counter current chromatography	*Juglans mandshurica* maxim	Antioxidant capacity	394 1,2,3,4,6-penta-O-galloyl-β-D-pyranglucose, 1,6-di-O-galloyl-β-D-pyranglucose, 1,2,3,6-tetra-O-galloyl-β-D-pyranglucose, 393 1,3,6-tri-O-galloyl-β-D-pyranglucose
Extrusion	Fruits/*Opuntia ficus-indica*	Antioxidant activity	Betaxanthin and Betacyanin
High hydrostatic pressure	Whole carrots	Antioxidants	Total xanthophylls, Carotenoids
Enzyme-assisted extraction (EAE)	Residues/*Vitis vinifera* L.	Antioxidant activity	Resorcinol O-coumaric acid and Gallic acid
Supercritical fluid extraction (SFE): CO_2	Aerial parts/*Porophyllum ruderale*	Antioxidant activity	Phytol, α-copaene and Isosafrole
Solid-phase extraction (SPE)	Seeds/*Triticum spelta* L.	Antioxidant activity	*p*-hydroxybenzoic acid, *p*-coumaric acid, caffeic acid derivatives, *trans-f*erulic acid
Ultrasound-assisted extraction (UAE) or sonication extraction	Fruit/ *Ustilago maydis*	Anti-diabetic, anti-inflammatory, antioxidant, immunomodulatory, hypo-cholesterolaemia, anti-hypertensive, anti-microbial	Essential fatty acids (oleic and linoleic)
Pulsed electric field (PEF) extraction	*Carya illinoinensis* (Wangenh. K. Koch)/oil.	Antioxidant capacity	γ-tocopherol, Polyunsaturated fatty acids and β-sitosterol
Ohmic heating	*Viride*/aerial parts, *Origanum vulgare* subsp.	Antimicrobial activity and antioxidant activity.	Thymol and 4-terpineol

Four Steps are Involved in The Extraction of Natural Products:

1. The solute diffuses out of the solid matrix.
2. The solvent dissolves in the solute.
3. The solvent is absorbed into the solid matrix.
4. The extracted solutes are then collected.

The properties of the solvent, the size of the particles in the raw materials, the ratio of solvent to solid, the temperature during extraction, and the duration of extraction will all affect the extraction efficiency.

Factors Involved in Extraction Process: The scientific community has researched extraction from solid matrices in great detail, and it is used in the food, cosmetic, pharmaceutical, and chemical sectors for a variety of reasons. The process typically entails contacting a solvent with a solid matrix containing the target molecules to extract the compounds that the solvent can solubilize under certain conditions. In addition to solubility, additional critical process variables include solvent flow, substrate physical characteristics, and extractor size. This section offers a summary of several process considerations that should be made when choosing an extraction parameter set and a solvent, or even when scaling up a thoroughly studied process. Particular emphasis is placed on Soxhlet apparatuses and high-pressure solvent extraction techniques [3].

Solubility: Every extraction method is based on the solubility of one or more components in a solvent, which can be either pure or a mixture of two or more compounds. First of all, it is clear that solubility depends on the solvents and the solutes chemical affinity; hence, choosing the appropriate solvent is crucial to achieving the desired outcome. For example, polar solvents frequently solubilize polar molecules, whereas non-polar solvents solubilize non-polar molecules. Furthermore, other elements of the extraction process, such as temperature and pressure, might have an indirect impact on solubility. It is well known that liquids make better solvents than gases, and solubility in extraction using compressible solvents (gases and supercritical fluids) is a direct function of solvent density. The solubility of these solvents can change significantly with these two process factors because pressure and temperature have an impact on their density, as demonstrated by both experiments and equations of state. In compressible solvents, solubility must rise with pressure and fall with temperature according to the solvent density [3].

Additionally, when the solute vapor pressure is taken into consideration, temperature can have a reverse effect on solubility. Raoult's law equation predicts that, as this property increases with temperature, the solubility or equilibrium concentration of solutes in the "gaseous" phase should also climb. This is a rudimentary version of the trend.

$$Y^* = \boldsymbol{xPvap}\,(\boldsymbol{T}) \,/\, \boldsymbol{P}$$

Where Pvap = vapor pressure of the solute (Pa); x = solid (substrate) phase equilibrium concentration (kg solute/kg solution); Y* is the solubility (kg solute/kg solvent) of the fluid phase equilibrium concentration; T is the extraction temperature (K); P is the extraction pressure (Pa).

In certain SFE processes, these contradictory effects of temperature on solubility are observed. Furthermore, temperature can alter the dielectric constant, which adjusts a solvents, polarity, and other solvent properties. This is crucial for pressurized water extraction processes. As a result, changing the process temperature may also affect a compounds solubility in a particular solvent [3].

Solubility is a key factor in large-scale operations for figuring out what drives mass transfer from substrate to solvent, particularly when taking economic factors into account. A crucial element of many mathematical models that describe extraction kinetics and mass transfer is the concentration gradient between the solvent phase and substrate surface. In actuality, equilibrium was formerly believed to occur on the substrate surface at equal concentrations and solubility of the solute. Since the concentration gradient increases as the solute concentration in the solvent phase decreases, low solvent phase concentrations are therefore preferable to ensure high mass transfer rates.

Utilizing high solvent flow rates can help maintain low solvent phase concentration by lowering residence time to a point where solute concentration is kept far below saturation. Nevertheless, this results in higher operational expenses for the preparation, recycling, and use of solvents. Therefore, even though mass transfer rates are being reduced, maintaining a reasonably high solute concentration in the solvent might be an example of operational economy. Scale-up requires taking these concerns into account by comparing the necessary extraction rates to the costs of purchasing, processing, and recycling the solvent.

Solvent Flow Rate: The choice of solvent flow rate has a major impact on the equilibrium relationships between the solvent phase and the thin solvent film involving the substrate, which can be thought of as a mass boundary layer. Consequently, the gradient between equilibrium and fluid phase concentration may be the main driving force behind the extraction process. The solute concentration in the extractor's solvent, which is established by the solvent flow rate, affects the driving force. Even when the influence of solvent flow rate is ignored, strong concentration gradients caused by exceptionally high solvent flow rates can speed up mass transfer in the fluid phase. Since intraparticle diffusion is in charge of this process, increasing the solvent flow rate would be pointless and incur additional costs. When the solvent flow rate is low enough to ensure that intraparticle diffusion keeps the solute equilibrium concentration near the solid surface, something happens. Next, the solvent velocity may have an impact on mass transfer and increasing it may be advised to quicken the extraction process. In these cases, solubility and fluid-phase convection govern the process [3].

Substrate Properties: Naturally, the characteristics of the source material affect extraction yields and kinetics. Therefore, in order to enhance yield and extraction rate, the substrate must be prepared for extraction carefully. In addition to drying, which is frequently advised to lower the product's unwanted water content, particle size control is a constant consideration. Particle size has a direct correlation with diffusive channel length in Fick's diffusion equations, which means that it influences mass transfer kinetics. In this regard, grinding the solid raw material to the lowest size feasible would naturally lower these barriers by removing obstructions to the solute solvent outflow from the particle pores and the solvents diffusion into the particle.

However, overly small particle reduction can result in compacting, which effectively implies that blocks of substrate will develop and the solvent won't pass through. We designated these as "preferential pathways." The extraction yield is decreased because the solvent inside these blocks does not come into contact with the solute. The bed porosity, which must not be unduly low, can also be controlled to limit the presence of preferential paths [3].

In terms of scale-up, the conversation needs to center on methods that can be applied on an industrial scale to both load the extraction columns with raw material and minimize particle size. This process phase is definitely a lot more complex than in the laboratory, where extraction columns are manually filled and procedure time is not a major concern. Two or more extractors are typically found in industrial extraction operations, with one extractor being reconditioned while the others are in use. The extraction bed packing needs to be completed within the extraction cycle time to avoid the formation of compact patches that would prohibit the solvent from flowing through them. For the extraction bed to be as uniform as possible, automated processes have to be used in this step [3].

Extraction Bed Geometry: The substrate that is positioned inside the column where the procedure takes place is known as an extraction bed. Its "geometry" is composed of the length (L) and diameter (D) as well as the ratio between these two dimensions. Because they are thought to be small enough to not be affected by spatial effects, laboratory-scale beds and columns are frequently dimensioned without worry. However, some consequences may arise in large-scale processes, which should be taken into account. Care must also be taken to prevent these effects and replicate the behaviour of the laboratory and pilot plants on a larger scale. Since extraction columns are typically positioned vertically, the solvent crosses the substrate in an upward motion parallel to gravity. Thus, mass transport and fluid dynamics may be impacted by the gravitational pull. Natural, or free, convection is the term used to describe how gravity affects mass transfer. Long extraction beds may show observable concentration gradients along their length since there is a pure solvent at the intake and a concentrated mixture at the output. Due to gravity influences and the increasing solution density with concentration, the extraction rate will decrease, and back mixing will occur. Long extraction beds are therefore not advised in all circumstances. Additionally, radial impacts are to be avoided. When there is an uneven solvent distribution at the column intake, they may manifest. Because laboratory columns have a diameter that is not significantly greater than that of solvent pipes, this problem is less evident. For industrial extractors, a distribution system is usually necessary to guarantee that the solvent is distributed uniformly over the substrate bed. Generally speaking, L/D ratios of five to seven are recommended for industrial extractors according to the axial and radial challenges that are mentioned [3].

Extraction Process for Dill Seed Oil: The Greek word aneeson or aneeton, meaning strong-smelling, is the source of the genus name Anethum. In Ayurvedic medicine, it is frequently used to treat colic, ease discomfort in the abdomen, and aid in digestion. Shatapushpa has several Ayurvedic properties, including katu tikta rasa, usna virya, katu vipaka, laghu, tiksna, and singdha gunas. It cures "vata," "kapha," ulcers, uterine pain, and eye disorders. Charaka advised using a mixture composed of a mixture of milk and crushed castor seeds, linseed, and shatapushpa (A. graveolens) externally for rheumatic joint swellings. The herb (A. graveolens) was said to have tonic, revitalizing, and intellect-promoting qualities by Kashyapa Samhitaa. It is a component of Unani medicine for gripe water, colic, and digestive issues. Anethum graveolens L. is used to make more than 56 ayurvedic remedies, including Dhanwan-thararishtam, Dasmoolarishtam, Mrithasanjeevani, Saraswatharishtam, Gugguluthiktaquatham, Maharasnadi Kashayam, Dhanwantharamquatham, and others. Dill is believed to be native to either South-East Europe or South-West Asia. It is indigenous to the southern USSR, the Mediterranean, and Central Asia. Since, the Egyptian era, anethum has been used as a condiment and medicine. Egyptian medics used it 5,000 years ago, and traces of it have been found in Roman ruins in Great Britain. It was believed to offer protection from witchcraft during the Middle Ages. The Greeks used dill leaves to cover their heads in order to fall asleep [4].

Experimental:

Process of Extraction of Dill Seed Oil: Hexane, dichloromethane, and methanol were used as distinct solvents in the Soxhlet extraction process to produce the various extracts from dill seeds. 500 mL of hexane, dichloromethane, and methanol were extracted from 100 g of powdered dill seeds in separate round-bottomed flasks in the hot tub. The crude extracts were kept in storage at 4 °C after the solvent was eliminated using distillation on a rotary evaporator. 20 mg of each chemical were dissolved in 10 ml of methanol to create the stock solution (2 mg/ml). Using methanol as the solvent, the stock solution was then diluted to the required concentrations of 1.0, 0.5, 0.25, 0.1, and 0.05 mg/ml [5].

TABLE 3: A FEW WORKS ON THE SCALE-UP OF EXTRACTION PROCEDURE

Sr. no.	Methods	Highlights	Reference
1.	Extractions Assisted by Ultrasonic	This investigation assessed the impact ofultrasonic-assisted extraction (UAE) onthe extraction of antioxidants, phenoliccompounds, and flavonoids from dill. The UAE raised the antioxidant activity of alldill extracts and enhanced the extractionyields of total phenolic components andtotal flavonoid content.	Khakhanang Ratananikom et al., (2022)[6]
2.	Steam Distillation	This study's goal was to look into the chemical makeup and antibacterial properties of the essential oil of dill fruit that was grown in Thailand, province of Udon Thani. Steam and hydrodistillation were used to extract the essential oils fromdill fruits. Compared to steam distillation, hydro distillation produced a larger yield of essential oil	A. Ruangamnart et al., (2015)[7]
3.	Maceration	Some gastrointestinal conditions are treated with Anethum graveolens seed (dill) as a folk medicine. Our objective was to assess antiulcer aqueous andethanolic extracts and anethumgraveolen's acute toxic effects in mice.	Hossein Hosseinzadeh et al.,[8]

Mechanism of Soxhlet Apparatus: The Soxhlet extractor, created by Franz von Soxhlet (1848–1926), is a well-known tool used in chemical, pharmaceutical, environmental, and food-related labs. It is utilized to batch-separate soluble components from an insoluble solid matrix by using a volatile solvent. Specifically, based on the polarity of the active components to be extracted, the Soxhlet apparatus operates with polar, semi-polar, or nonpolar solvents. This makes it possible to extract "active compounds of interest" from solid waste, whether it's wet or dry, using a heated solvent. The main benefits of the Soxhlet process include full extraction and low solvent consumption; however, heating is necessary for solvent boiling. These days, the Soxhlet apparatus might be used to test "greener" biobased solvents for more environmentally friendly extraction procedures, such as R-limonene and 2-methyltetrahydrofuran, which are derived from crop by products [3].

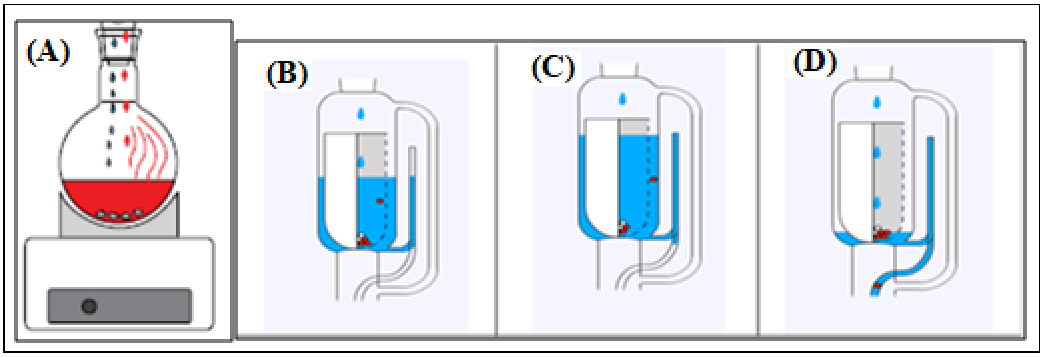

FIG. 1: ILLUSTRATION OF THIMBLE PROCESSING USING WASTED SOLIDS AND SOXHLET EXTRACTION

A. While some condensed solvent returns to the heated bottom flask as droplets, the solvent boils in it;

B. As the solvent seeps out of the solid sample and the paper thimble, a lateral arm permits the solvent vapors to rise; the solvent is recondensed by the refrigerant.

C. The extracted compound solvent fills the extraction chamber below the siphon height;

D. Siphoning is activated, causing the extract solution to descend into the bottom flask, where, the extracted solids build up in the solvent cycle after cycle.[3]

Describe Scale UP: The term "scale up" refers to the process of creating large-scale machinery or a full-sized plant (prototype) utilizing information gathered from laboratory tests.

Objectives of Scale-up:

- To offer a master manufacturing formula.
- To determine the essential components of the process.
- Process evaluation, validation, and finalization.
- Producing physically and chemically stable products.
- Reviewing processing equipment and developing production and process control guidelines.

Producing an equivalent process outcome at a higher production rate than previously achieved is known as "scale-up" [3].

Here, we examine this definition in detail.

1. "The Previously accomplished" refers to a procedure that is already known since it was carried out and its outcomes were attained on a tiny, analytical scale, which is more generic. Scaling up necessitates prior knowledge of the process.
2. Data collected during the small-scale process, including yield, product quality, and chemical composition, are referred to as "an identical process result." This suggests that throughout scale-up, the same standards should be met with the least amount of modification required, if any.

3. "A larger production rate" means that, in comparison to what was achieved on a small scale each hour, day, or year, the same product that was made on a small scale needs to be produced on a large scale. Additionally, "how much" higher certain rates should be can be determined; this is referred to as the "scale-up factor."It is important to stress that we are considering the fact that increasing output rates is necessary when using larger or even more equipment than in small-scale production. The large-scale unit's overall capacity will be larger in both scenarios. Therefore, it is essential to estimate the large-scale equipment requirements in order to maximize the intended production rate. The link between these two variables, or the scaleup, is the central idea of the scale-up process standard. The scale-up concepts can also be approached from a thermodynamic perspective.

The features of a system are classified as extensive or intensive in classical thermodynamics. Mass, volume, and energy are examples of extensive qualities that are dependent on the size of the system, whereas concentration, density, and viscosity are examples of intensive attributes that are not. The goal of scale-up is to increase the "extensive data" in compliance with appropriate criteria while preserving the values of the bulk of the process's "intensive data."

Upscaling Dill Seed Oil Extraction Procedures: Using additional solvents will speed up the dill seed oil extraction process. Plotting the extract yield or the mass ratio of the extract to the solid feed, against time or even the amount of solvent used can be used to generate an extraction curve common at the analytical scale.

Hence, the goal of scale-up is to replicate the exact same curve at a larger scale by maintaining certain of the analytical scale's intensive extraction parameters and increasing others according to predetermined standards. The following intensive parameters may need to be maintained during extraction processes:

- Temperature (T)
- Solvent
- Extraction of oil

Lastly, it's important to determine the outcomes that should be replicated, including:

- ✓ Yield of extraction (X0);
- ✓ Extraction velocity (mass transfer rate);
- ✓ Extract quality (flavor, aroma);
- ✓ Chemical and physical qualities of the extract (density, composition, viscosity, etc).

The curve can be used to determine the extraction yield and velocity among this data; further testing and measurements are needed to assess the remaining data [3].

Scale UP Criteria for Dill Seed Oil: When the product and its necessary attributes are clearly understood and the small-scale actions required to produce it are well-established, a process can be described. It allows for the definition of 1st and 2nd scale-up criteria.

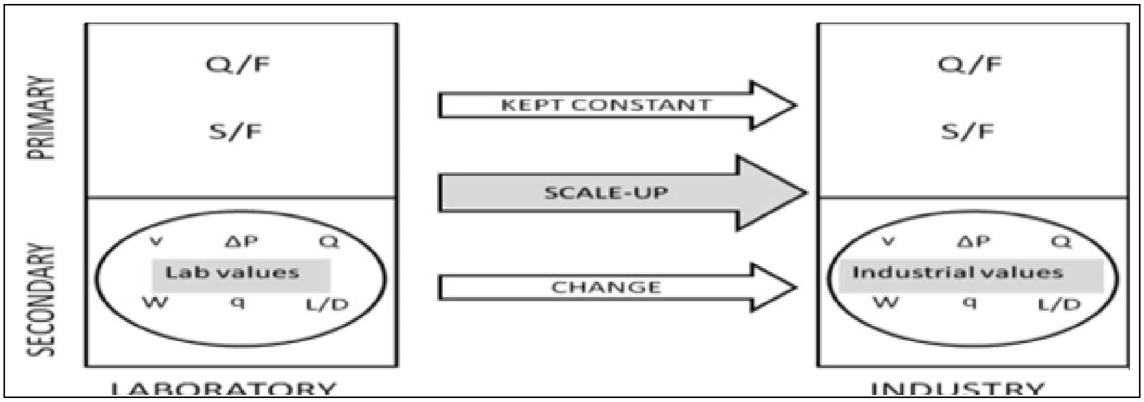

FIG. 2: ILLUSTRATION OF 1ST AND 2ND EXTRACTION SCALE-UP CRITERIA. F¼ solid feed; S¼ solvent mass; Q¼s solvent flow rate; v¼ solvent velocity; DP¼ friction loss; q¼ heat transfer rate; L¼ extractor length; D¼ extractor diameter.

Primary Scale-UP Criteria for Dill Seed Oil: A fundamental scale-up criterion is a process parameter, or even a set of process parameters, that, at any scale, yields the desired process result, *i.e.*, the product and its attributes. Thus, those settings must be followed precisely when executing scale-up. The outcome could change if certain process factors are adjusted. Finding the factors that, when conserved, ensure the same outcome is the next hurdle in scale-up. The aim is to obtain a product with a precisely determined chemical composition and yield before beginning the extraction procedure. Viscosity, temperature characteristics, flavor, scent, and a variety of biological activities are a few examples of features that can be added to the desired result for more specialized uses. The solvent velocity, pressure, and temperature are the main scale-up requirements. Theoretically, these three values can be maintained at the same values at all scales [3].

Secondary Scale-UP Criteria for Dill Seed Oil: A process may undergo certain mechanical and physical alterations as a result of scale modification. As these effects cannot be completely eliminated, they should at least be thoroughly understood so that the process engineer may use the primary scale-up criteria to regulate them and, as a consequence, get the desired outcome. The secondary scale-up criteria outline how those modifications must be implemented. These are a few instances of alterations brought about by increased extraction scale.

Heat Transmission: which is required to achieve and sustain the temperature during the extraction process (keep in mind that temperature is one of the primary components). Temperature-controlled analytical-scale extraction experiments can be carried out with simple instruments such as electrical resistances, thermostatic baths, or even by placing the extractor inside an oven. Furthermore, because the heated volume's internal thermal resistance is within the range of cubic centimetres, it can be easily overcome. Otherwise, an industrial extraction column that may have more than one cubic meter cannot be heated using the small-scale heating devices. Hot water or vapor lines are required, and the heating fluid often travels through a jacket that has the extractor attached to it. As a result, when scaled up, the heat-transfer processes alter. Since longer pipes make it impossible to obtain the same level of process control as in analytical tests, it is really expected that heat loss on a large scale will be proportionately higher. This indicates that more heat will be lost to the environment from the extractor. A thorough investigation into methods to enhance heat transfer during the extraction process is necessary in light of all these modifications to the process features. Naturally, this growth cannot simply be proportionate to the extractor's rise [3].

Solvents of Distribution: The basis of all extraction processes is the contact of a solvent with a substrate from which the target compounds, or extracts, are extracted. The solvent must then come into contact with all of the solid feed, which requires a well-designed procedure. An extractor, with the arrows signifying the solvents flow stresses as they enter. It should be noticed that some substrate sections toward the bottom of the extractor wall are not touched by the solvent strains because of insufficient dispersion at the input. If the solvent does not reach all of the substrate, some of the soluble compounds will remain unextracted, and the yield will be lower than expected. There must be systems in place to stop this phenomenon and encourage the solvent's uniform distribution, because it is undoubtedly undesirable. These mechanisms might be small-scale or large-scale in nature. For instance, inert glass spheres can be utilized in the lab at the extractor input to create an area where the solvent can spread evenly along the column radius.

However, the industrial-scale unfeasibility of glass spheres means that alternative methods of dispersing the solvent flow must be found, which may involve developing new accessories. One more secondary scale-up criterion is required to specify the manner in which the uniform solvent distribution will be scaled-up. The positioning of the substrate inside the extractor is another issue that could have an impact on the solvent dispersion. Although such work is done manually at the analytical scale, this phase needs to be mechanized in an industry. The key challenge in this procedure continues to be maintaining the extraction bed consistent, that is, with the same apparent density and porosity throughout all of its sections along the extractor.

For small-to large-scale operations, maintaining porosity is crucial to preserving the same flow pattern since the solvent will pass through the extraction bed's empty volume. Furthermore, too much compaction of the substrate could cause the solvent to establish preferential pathways that might not reach all of the extractable material. If the extractor is manually fed, the operator can control this parameter by varying its strength. However, this can be a big issue in an industrial setting, thus a secondary criterion is definitely important.[3]

Mass Balance: The material flows in accordance with the general conservation law forthe construction of simplified mass balances:

Input (at Soxhlet start up) = Output (at Soxhlet test conclusion) the following material balance equations were written, correspondingly, for dry solids (DS) the solvent (S) and water (w):

$$xS, SOLV*WSOLV = WS,E + WS,SP + WS,L$$

$$xw,TS*WTS + xw, SOLV \cdot WSOLV = Ww, E + Ww, SP + Ww, L$$

$$xDS, TS \cdot WTS = WDS,SP + WDS,E$$

where, as determined by weighing either at the start or completion of the test, WTS, WSOLV, WDS, and SP are the masses of the wasted biomass (SP), the raw sample, the solvent, and the dry solids, respectively. The weight differential between the test's conclusion and the mass of water and solvent in the spent biomass under the hood drying is determined after two days:

$$WDS,E + Ww,E + WS,E = \text{known value.}$$

Finally, the total mass (E) of the extracted solution can be computed using the difference in the flask's weight at the end of the test.

$$WDS,E + Ww,E + WS,E = \text{known value.}$$

Should account for the potential for vapor loss due to incomplete condensation in the Graham condenser. They therefore designated WS,L for the solvent mass and Ww,L for the water mass lost as the unknown variables. Should consider the composition of the liquid phase that was losing through evaporation at the end of the test after being wetted with depleted biomass (SP) and allowed to dry for two days under the hood. The composition of the liquid phase and the extracted solution (E) is almost identical. The resulting two equations are as follows:

$$Ww,SP/(Ww,SP + WS,SP) = Ww,E/(Ww,E + WS,E)$$

$$WS,SP/(Ww,SP + WS,SP) = WS,E/(Ww,E + WS,E$$

The following congruence equations simplify to two identities:

$$xS,SOLV + xw,SOLV = 1; 2)xDS,TS + xw,TS = 1$$

While the mass fractions related to the fresh solvent xS, SOLV e xw, SOLV are known from its specification sheet, the mass fractions related to the tested biomass (TS) xDS, TSe xw,TS are known from the Kern moisture analysis.

NE = 7 is the sum of the previously mentioned equations.

The number of unknowns, Ww, SP WS, SP WDS, E Ww, E WS, E WS, L Ww, L, is indicated by the notation NV = 7.

The degrees of freedom are therefore represented by the quantity NGL= NV - NE = 0 [3].

TS	Raw biomass (Test Sample)
SOLV	Fresh solvent (SOLVent)
E	Extracted solution
L	Evaporation Loss
SP	Depleted biomass (SPent solids)
S	Solvent
------	Recirculation streams

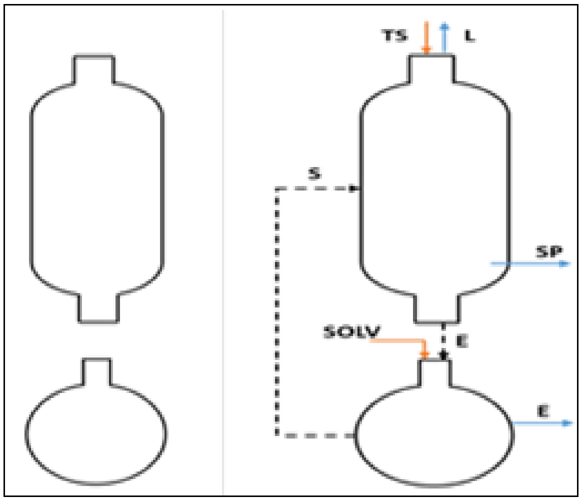

FIG. 3: REPRESENTATION OF (A) THE SOXHLET APPARATUS AND (B) THE ASSOCIATED MATERIAL FLOWS

Energy Balance: The latent heat of the evaporated liquid volume plus any heat dispersion over a cycle establishes the amount of thermal energy that must be transferred to the bottom flask via the bottom warmer.

$$QHEATER = QEVAP + QDISP$$

The clarifications that follow:

i. QDISP = 0 indicates that there are no heat losses;

ii. The pure solvent evaporates at room temperature. As a result, the equation before becomes

$$QEVAP = V\ \rho SOLV\ \lambda solv$$

Where, V is the volume of solvent evaporated in a single cycle [m3], ρSOLV is the solvent 's density [kg/m3], and ▫SOLV is the latent heat of vaporization at atmospheric pressure [J/kg].

The total energy needed for boiling during the full Soxhlet test can be obtained by multiplying the number of cycles.

$$QTRANS = WSOLV\ cp,SOLV\ (Tb - Tamb)$$

Where, cp,SOLV is the solvent's specific heat [J/(kg K)] at atmospheric pressure.

The volume V of solvent that evaporates in a single cycle in the aforementioned equations only represents a portion of the total solvent in the flask, which has a mass WSOLV. A technique for measuring the real volume sucked in cold conditions, which precisely matches the solvent's evaporated volume. After that, V = 70ml was discovered experimentally [3].

CONCLUSION: In conclusion, using co-solvents in Soxhlet extraction offers a viable way to improve the solubility of important ingredients in dill oil. In addition, the incorporation of ultrasonic-assisted extraction (UAE) has been demonstrated to be a highly effective method for enhancing the extraction yield of flavonoids, phenolic compounds, and antioxidants from dill. The focus of this study is on the chemical makeup and antibacterial properties of dill fruit essential oil, which is grown in the Thai region of Udon Thani. This emphasizes how important extraction techniques are in determining yield and bioactivity. A comparative study showed that dill fruits produced a significantly larger amount of essential oil by hydrodistillation than by steam distillation. These results highlight how crucial extraction methods are to maximizing the output of essential oils, offering insightful information to sectors that depend on dill essential oil for a range of uses. Furthermore, research into the potential medical benefits of extracts from *Anethum graveolens* seeds, notably in the treatment of gastrointestinal disorders, highlights the continued value of herbal medicines in traditional medicine. A thorough knowledge of the pharmacological profile of dill seed extracts is aided by the examination of acute toxic effects in mice and the assessment of aqueous and ethanolic extracts for antiulcer activity. Overall, this study highlights the potential of dill as a source of bioactive chemicals with medicinal implications and advances our knowledge of dill oil extraction methods. The findings show the importance of dill extracts in both conventional and modern medical practices, opening the door for more research into the wide range of uses of dill extracts in pharmaceuticals, nutraceuticals, and other fields.

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