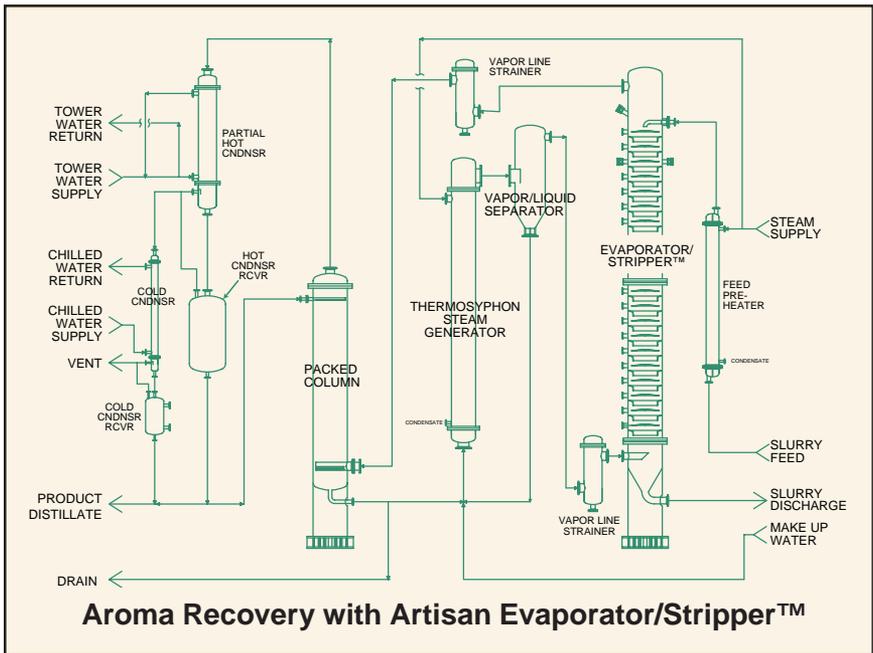


Process Solutions Handbook

Food, Ingredients, Edible Oils and Nutraceuticals Applications



73 Pond Street Waltham, MA 02451-4594

781-893-6800 fax: 781-647-0143

info@artisanind.com

www.artisanind.com



All things change except for the love of change.

Anon, (Madrigal, 1601)



Artisan Industries Inc

Process Solutions Handbook

This *Process Solutions Handbook* has been created to stimulate new processing ideas and address the need for more creative and competitive approaches to processing challenges. As a business group, foods, ingredients, edible oils, cosmetics and nutraceuticals manufacturers face many similar production and processing issues. Today, more than at any time in the past, growth and competitive pressures challenge this industry group globally. By providing unique answers to these challenges, Artisan has witnessed the increasing pace and more demanding nature of the solutions sought by these industries. Artisan's many years of participating at the leading edge of process development has given us the experience and now the opportunity to share many innovative processing ideas and solutions.

Evaporator/Stripper is a trademark and , Jet-Vac, Dualflo and Rotothem are registered trademarks of Artisan Industries Inc.

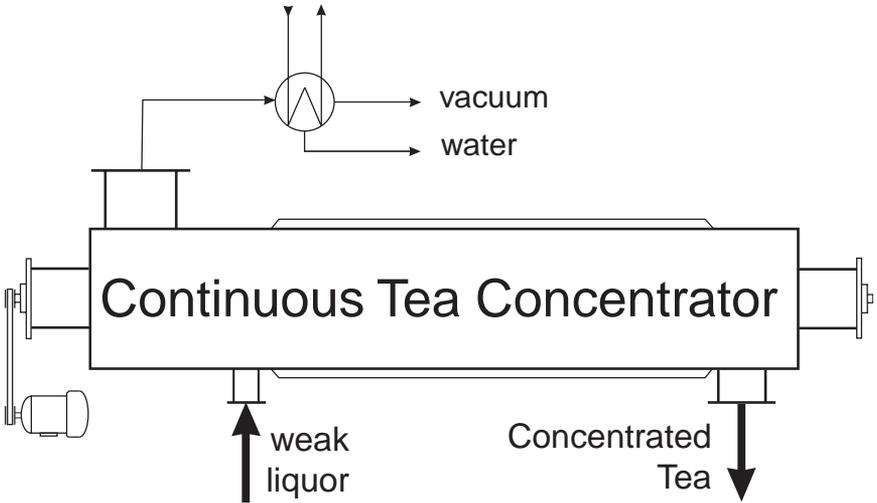
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Continuous vacuum concentration of tea from 15% solids to 50%. Concentration is carried out at 300 Torr and 170° F.



Concentrating Beverages

Problem: Concentrating coffee, tea, and other solids containing liquids pose severe operating challenges to the coffee, and beverage industry. Typically, conventional technologies, such as multiple effect, falling film, or forced circulation evaporators are used to accomplish this task, with mixed results depending on the particular application, and the degree of solids concentration. These problems generally stem from the heat and shear sensitivity, to foaming, and fouling characteristics of these materials.

Solution: Use the Artisan Rototherm® mechanically aided thin film evaporator to overcome these problems, and concentrate in a single pass a variety of beverages to solids contents, typically achieved in two or more unit operations. The Rototherm's ability to concentrate from a dilute feed to very high solids content, without product degradation, makes it the most versatile processor in the industry. The combination of short residence time, highly turbulent film, and the robust design of the unit, enables uninterrupted operation longer than any other processor. Due to the horizontal design, and close clearance between the spinning rotor and the inner shell, CIP is generally accomplished by charging the vessel with a few liters of suitable cleaning solution, and running the machine for a few minutes, drastically reducing consumption, and waste generation.

Examples:

- Concentrating coffee to greater than 80% solids, in a single step, achieving a consistent high quality concentrate.
- Concentrating caffeine from a wet solvent rich solution to a dry powder, with less than 100 PPM residual solvent in a single step.
- Concentrating tea from a dilute solution to greater than 50% solids in a single pass, without product degradation.

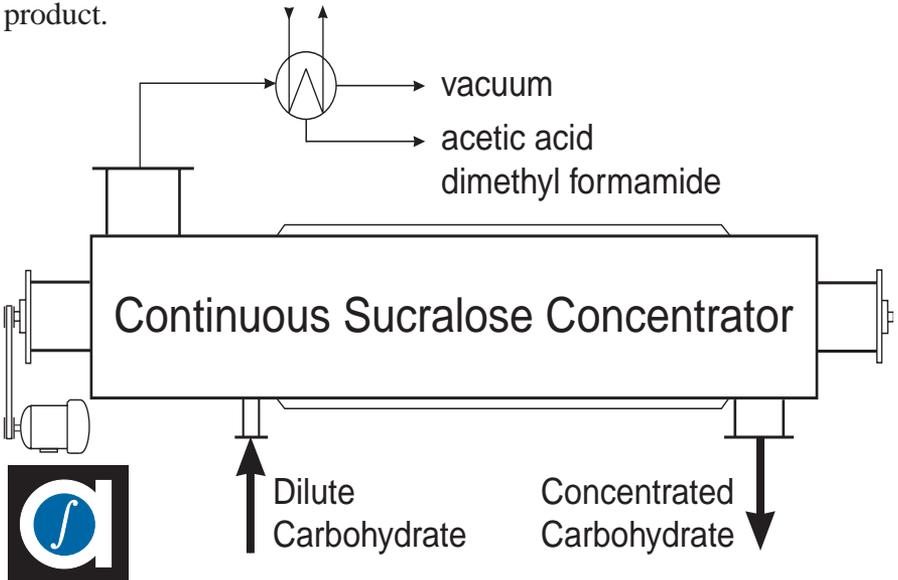


Concentrating Carbohydrates

Problem: Concentrating carbohydrates, and other similar heat sensitive products can not be accomplished by conventional evaporation technologies. The extreme sensitivity of these materials to time and temperature exposure, further compounded by foaming, and fouling tendencies, make them particularly challenging to process without product degradation, and fouling of the heat transfer surfaces.

Solution: The Artisan Rototherm® mechanically agitated evaporator is uniquely qualified for these types of applications, owing to its extremely short residence time, generally measured in seconds, narrow residence time distribution, rapid surface renewal, and high heat transfer rates. The Rototherm® is able to evaporate in a single pass, up to 99% of a feed solution, while achieving turn down ratios of 10 and higher, without product burn-on.

Example: Continuous vacuum evaporation and concentration of an artificial sweetener (sucralose) from 16% solids to 73% solids by evaporating 93% of the solvent consisting of dimethyl formamide, acetic acid and cyclohexane, producing a highly concentrated carbohydrate product.



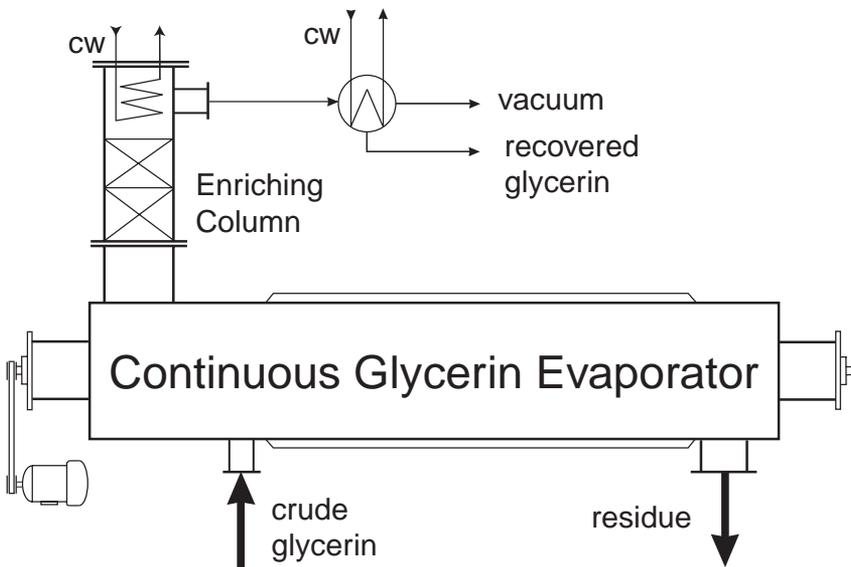
Recovering Glycerin

Problem: Recovering glycerin and other heat sensitive, solids containing valuable food additives can not be effectively accomplished using conventional evaporation techniques.

Solution: Use an Artisan Rototherm® mechanically agitated thin film processor to continuously evaporate and distill glycerin and other heat sensitive solids containing products, without color formation, while achieving greater than 95% yield. Owing to its extremely short residence time, narrow residence time distribution, rapid surface renewal, and high heat transfer rates, product degradation is minimized, while purity and yield are maximized.

Example:

- Recovering high purity glycerin from a vegetable oil, containing 20% glycerin and 2% sodium salts. Glycerin is distilled overhead, leaving a residue stream containing the oil, along with the salt.



Continuous vacuum evaporation/distillation of glycerin from a vegetable oil, containing 20% glycerin along with 2% sodium salts. High purity glycerin is recovered overhead while a concentrated residue is discharged as waste.

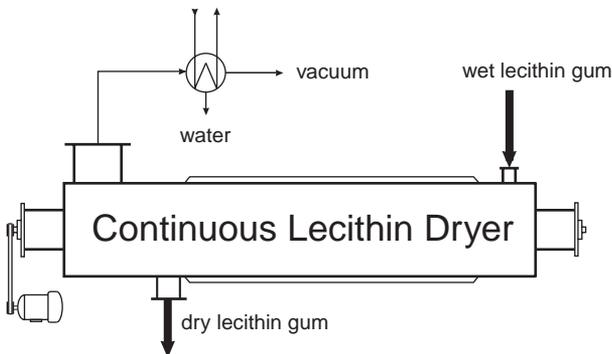


Drying Heat Sensitive and Viscous Food Additives

Problem: With conventional technologies it is extremely difficult to dry shear sensitive and viscous food additive products, such as lecithin. These highly viscous materials produce extremely low inside film coefficients in the laminar region, necessitating a mechanically agitated thin film surface to create turbulence; and rapid surface renewal to obtain higher inside film coefficient. Additionally, temperature sensitivity limits the driving force (the temperature difference between the heating medium and the process) resulting in larger surfaces and longer residence times, both of which increase the risk of product degradation.

Solution: Use the Artisan Rototherm® thin film evaporator for your most challenging drying applications. The Rototherm offers superior heat transfer, negligible pressure drop, and is able to process foaming, fouling, and solids containing materials with ease. Unlike vertical wiped or thin film evaporators that generally need an internal, or in some cases an external, entrainment separator device, the Rototherm, owing to its higher diameter to length ratios, and lower vapor velocities, does not require an entrainment separator in most applications.

Example: Continuously drying lecithin from a 50% solution to less than 1% moisture without product degradation.



Continuous vacuum drying of lecithin. The water evaporated is condensed and reused in the plant. Dry lecithin (less than 1% moisture) is pumped and immediately cooled to prevent degradation.

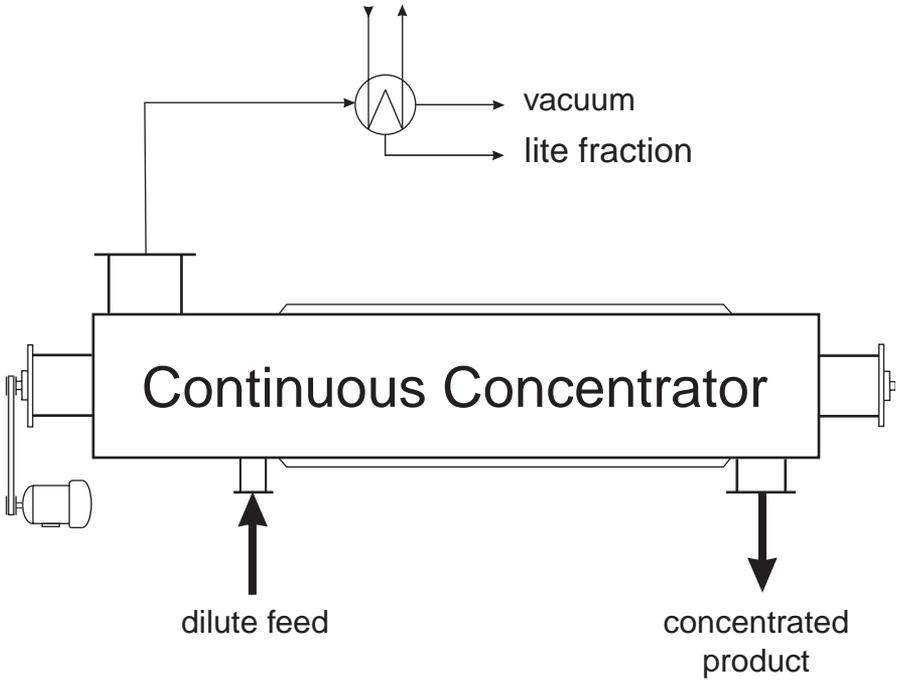
Recovering heat sensitive API's, intermediates or fine chemicals from solvent

Problem: Many compounds are produced in dilute solutions of solvent or are purified in mobile solvent streams, as in the case of chromatography. Heat sensitive, biologically active or intermediate structures within solvent laden, foaming, and/or fouling solutions are challenging mixtures for the recovery of valuable pharmaceutical or fine chemical molecules. Heat sensitivity presents a critical problem because elevated temperatures and exposure time must be controlled to preserve molecular integrity. Solutions with difficult compositions may foul heat transfer surfaces or form disruptive foams that inhibit the process. If heat transfer surfaces foul, throughput is reduced.

Solution: The Rototherm horizontal processor overcomes the problems of fouling, foaming, heat sensitivity, solvent content, etc. It allows concentrating and isolating the important ingredient quickly, often in seconds, in a single step. The process can be turned down to accommodate variable processing rates. The process is scalable to any capacity, and active compound efficacy can be demonstrated easily at small volumes and very low concentrations. The combination of vacuum operation and short residence time prevents product degradation. The unique rotor in the Rototherm processor allows for efficient self-cleaning of the internal heat transfer surfaces. The highly turbulent thin film at the heated surface enables heat flux rates of 10,000 to 50,000 BTU/hr-ft². Many feed and processing strategies can be employed to optimize the operation.

Applications: Active Pharmaceutical Ingredients (API), specialty chemical compounds.





Concentrating Biological Solutions

Problem: Concentrating heat sensitive food or biological solutions can result in poor and inconsistent product quality due to high heat history inherent in conventional technologies such as batch stills, rising or falling film evaporators, and natural or forced circulation evaporators. Nearly all these techniques require product recirculation to achieve high concentration levels.

Each technology poses a different and unique set of challenges to the design and operating personnel. These are some of the shortcomings of these technologies.

- poor heat and mass transfer capability
- high pressure drop
- long residence time
- turn down limitation
- product degradation/color formation
- inability to handle foam
- high fouling tendencies
- down time & loss of production
- concentration limitation
- yield limitation
- high head room requirement

Solution: Use the Artisan Rototherm® mechanically agitated thin film evaporator to overcome all of the above limitations, and benefit from the unparalleled longevity and rugged design of the Rototherm.

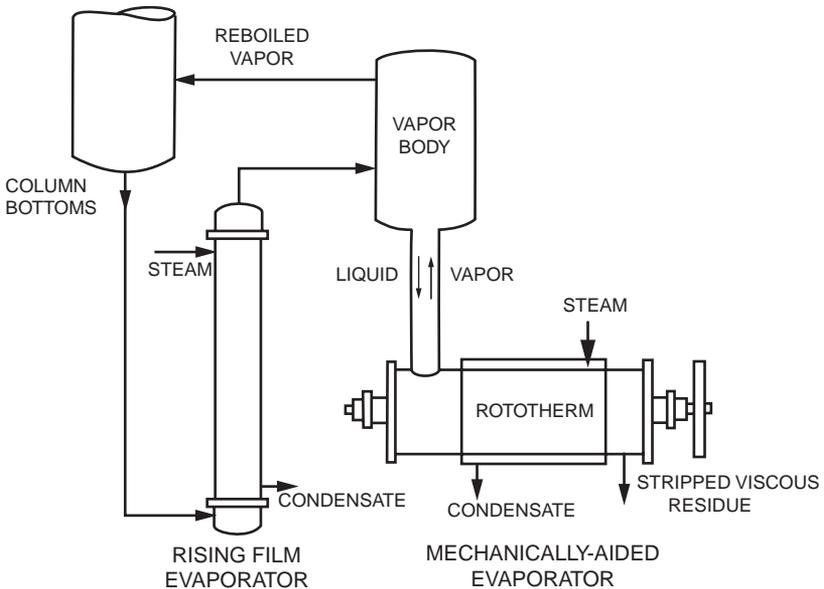
Examples:

- Concentrating a biomass (insecticide) containing 70% moisture, and producing a free flowing concentrate of high density solid particles (powder) with less than 10% moisture.
- Concentrating a fatty, aqueous salt solution from 20% solids to a relatively dry residue (no free standing moisture) containing no more than 10% moisture without product degradation. The process is carried out under vacuum to reduce operating temperature and minimize product degradation.



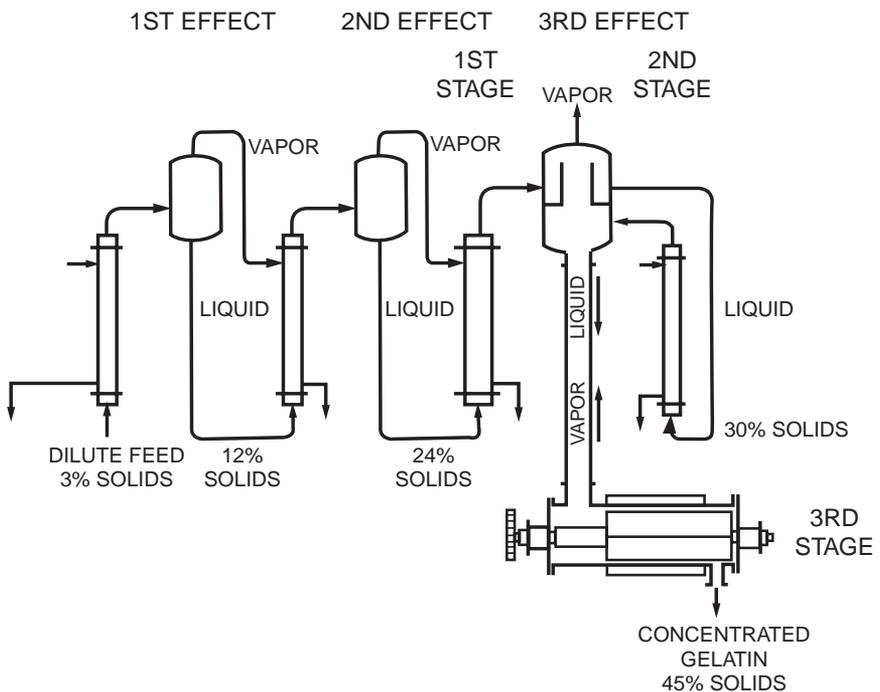
The Rototherm® as a Reboiler

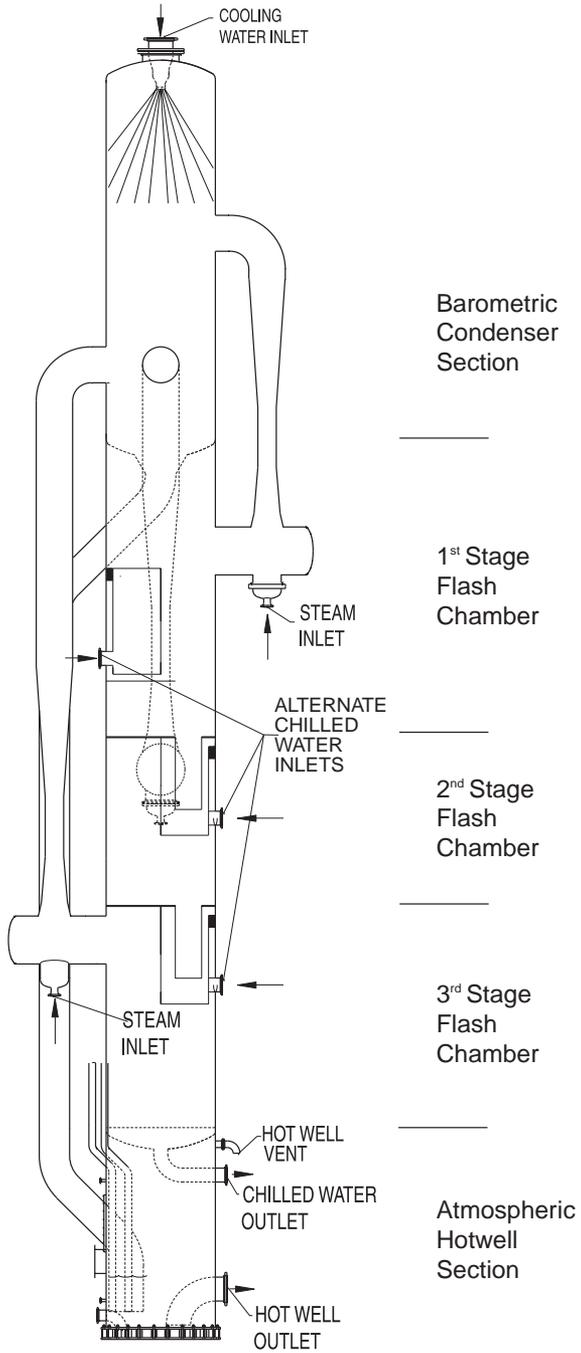
This flowsheet shows a horizontal mechanically-aided evaporator used in conjunction with a rising film evaporator as a reboiler at the bottom of a low pressure distillation column for processing a viscous, high molecular weight glycol. A stand alone rising film evaporator is not capable of removing all the volatiles from the bottoms material due to increasing viscosity. In general, rising film evaporator performance decreases quite markedly in that heat transfer coefficients decrease and pressure drop increases as fluid viscosities exceed 100 cps. Thus a forecut is taken in the rising film evaporator and the residual volatiles are evaporated in the Rototherm which is ideally suited to processing viscous materials.



Dehydrating Gelatin

This flowsheet illustrates the technique of multiple staging of rising film evaporators. It has been found that heat transfer performance in these units tends to decrease noticeably as the percent evaporation required increases beyond some critical value. The additional evaporator tube length required has a disproportionately adverse effect on pressure drop which in turn restricts the application of a single pass type unit. In these instances, it is more economical, both in terms of capital equipment costs and increased yield losses, to use a pre-evaporator to do the bulk of its evaporation in a forecut and effect the final concentration in a small, second stage evaporator.





Chilled Water without Refrigeration

Problems:

- a) chilled water is required in large quantities, or
- b) a process fluid needs cooling.

Considerations:

- the potential danger and expense associated with mechanical refrigerants (CFCs).
- the high maintenance, large space (real estate) requirements, and high operating cost (KwH) associated with mechanical chillers.
- a cooling tower that can not deliver a consistently low fluid temperature.
- ground or surface water sources not viable choices.

Solution: The Jet-Vac® chiller system produces chilled and/or deaerated water without refrigeration, and has the following advantages:

- no refrigerants
- no moving parts
- low operational cost
- unlimited quantities of chilled water
- small footprint

The principle of the evaporative cooling process is applied to cooling water or directly to the process fluid to achieve a low temperature and/or deaeration. A steam source at the site furnishes the motive force. A wide range of temperature differences or flows is possible.

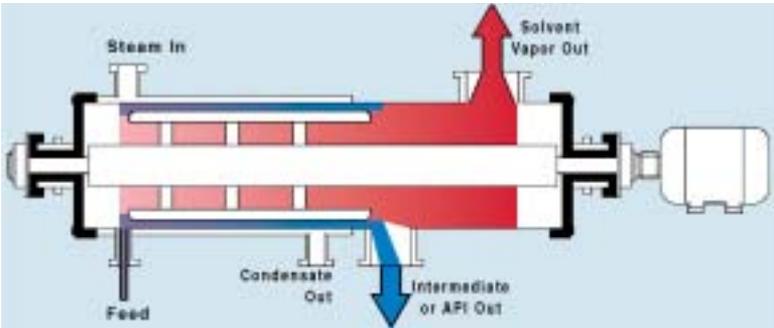
Examples:

Chilled Water			Cooling Tonnage	Tower Size (inch Dia x Ft tall)	Steam		Cooling Water	
GPM	°F out	°F Rtn			psig	#/HR	GPM	°F in
6000	74	84	2500	126x120	100	27,000	11,000	85
4000	68	81	2200	100x110	25	27,000	6,500	87
2000	75	85	835	66x62	180	11,000	4,200	90
1200	60	90	1500	78x75	15	35,000	7,000	90
200	40	59	160	42x50	135	3800	700	80
125	50	68	100	30x50	70	2500	600	90





Installed in a cGMP facility, this Rototherm is designed to manage solvents and to purify API, intermediates or fine chemicals.

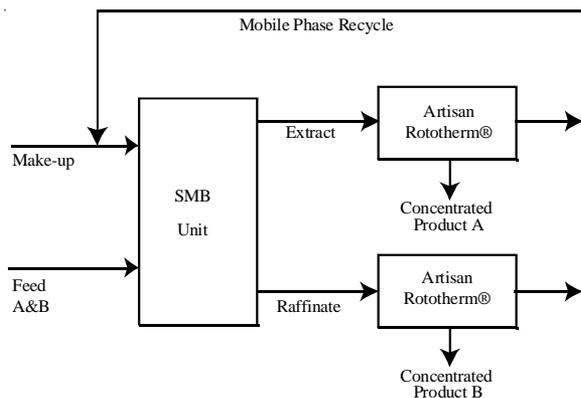


Managing Solvents in Chromatographic Separations

Problem: Separating compounds using single column prep LPLC, HPLC, or multi column SMB generates significant quantities of solvent from which the separated compounds must be quickly and economically recovered. Traditional batch glass rotary evaporators become unreasonably large and risky at the sizes necessary to handle the solvent volumes associated with batch prep chromatography and continuous SMB.

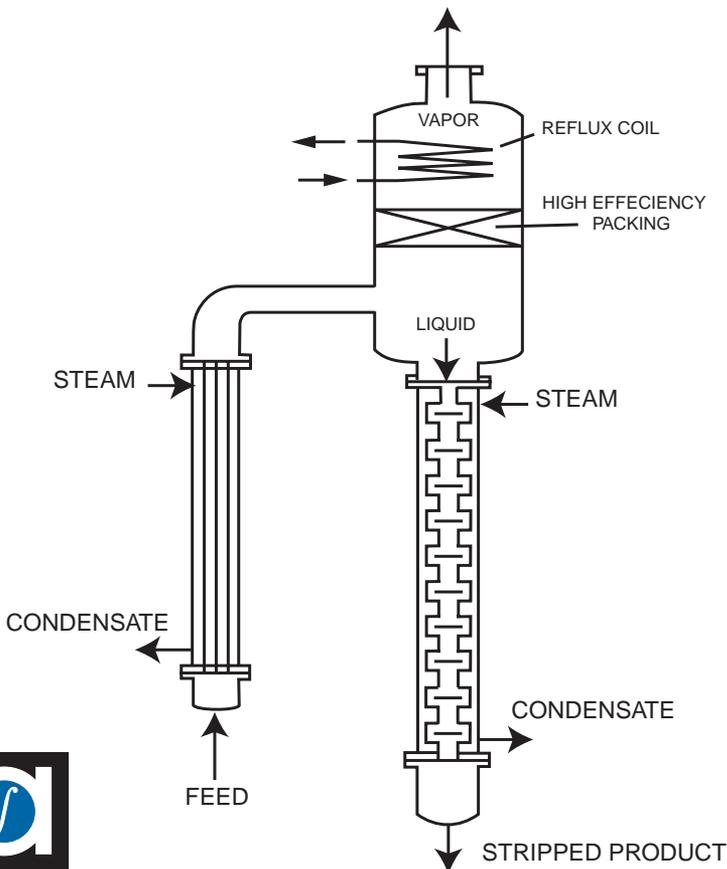
Solution: Artisan mechanically agitated thin film evaporators are being used instead of glass rotary evaporators. The short residence time is ideal for recovering heat sensitive products from the solvents. The low liquid hold up minimizes safety risks due to the short exposure time of the solvent at elevated temperatures. Finally, the wide range of capacities, and extremely high turn down and evaporation capability provide the processing flexibility required in typical laboratory or pilot environments.

Examples: Two Artisan mechanically agitated thin film evaporators are in operation for product recovery and solvent recycling at an SMB contract manufacturing facility. The evaporators are validated for cGMP operation.



The Evaporator/Stripper™ as a Rectifying Column

This flowsheet shows a rising film evaporator used in conjunction with the modified tube and disc stripper-low pressure drop packing combination as a rectifying column. This combination is used for instance in removing monomer from dimer and trimer fatty acids. The rising film evaporator is used here to do the bulk of the evaporation work in a simple, inexpensive piece of equipment so that the more expensive heat transfer area in the evaporator/stripper is used only where necessary. Overall yields for this system are in excess of 95%.

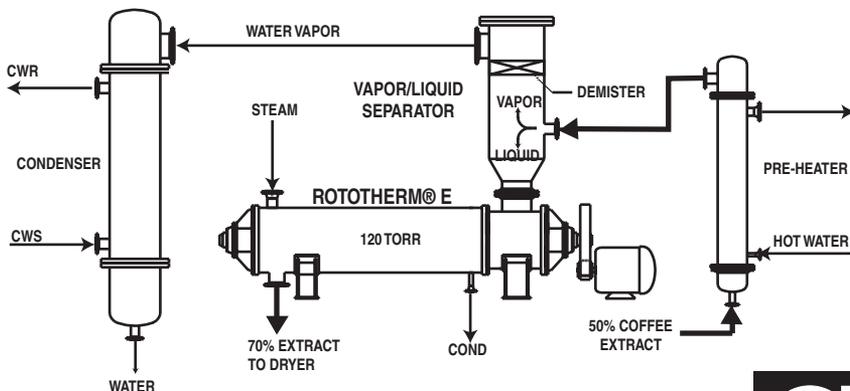


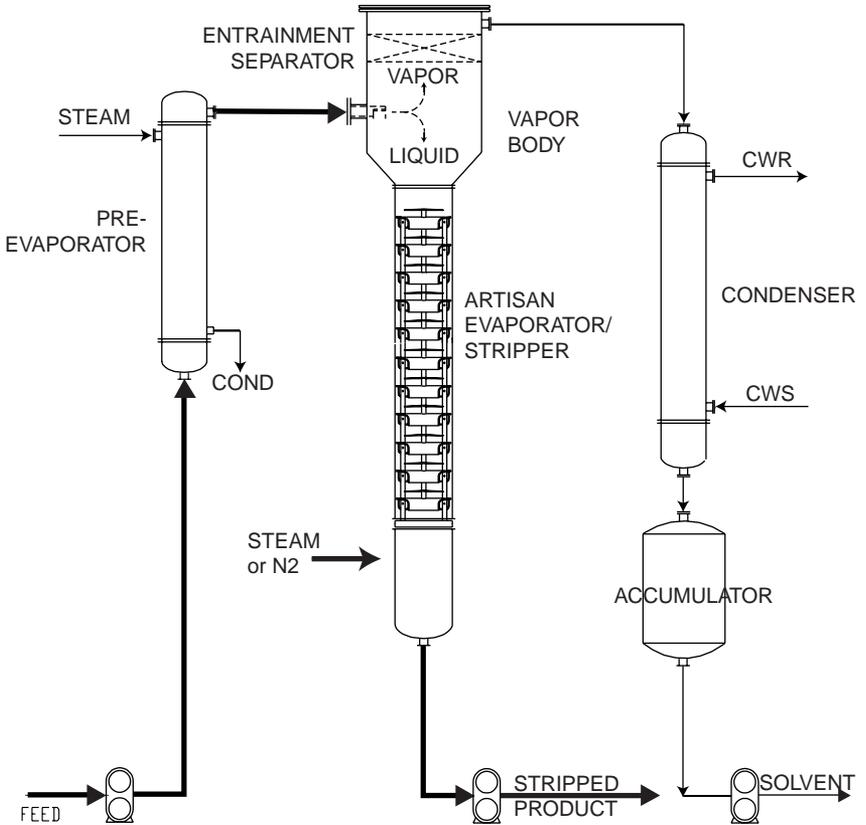
Energy Saving Strategies

Problem: Powdered beverages (instant coffee and tea, dairy beverages, fruit and vegetable juices & purees) and powdered ingredients (soy and whey protein isolates) are typically produced by pre-concentrating the feed material by conventional methods, such as membrane processes and non-mechanically aided evaporators, to reduce the evaporative load to the spray dryer. However, for most applications, these technologies cannot produce concentrates with optimal solids content.

Solution: Save energy and minimize loss of valuable aroma by using the versatile Artisan Rototherm® thin film evaporator to concentrate to 60% - 90% solids (depending on the type of product) prior to spray drying .

Example: Coffee processors typically pre-concentrate the 15-30% extract to 30-50% prior to drying. In the process below 50% extract (at a rate of 10,000 PPH) is concentrated to 70% solids. This reduces the load on the spray dryer by 1715 PPH, saving \$200,000/yr [based on 8000 hr/yr and steam at \$8/1000lbs]. In addition, the highly concentrated extract retains more of its natural aroma, thus yielding a superior product.





Desolventizing Edible Oils

Problem: Desolventizing and stripping organic solvents to very low (50 PPM or less) residual levels from concentrated vegetable oils containing emulsifying agents, such as lecithin. Typically, these separations are carried out in wiped film evaporators (WFE's), or packed columns. However, WFE's are single stage devices with limited mass transfer capability, requiring multiple passes; whereas, packed columns offer excellent mass transfer, but can not handle the viscosity.

Solution: Use Artisan's unique falling film disc and tube Evaporator/Stripper™, preceded by a rising film evaporator to remove the bulk of the solvent and strip the oil to low PPM residual level by using either stripping steam, nitrogen, or both depending on the desired moisture content of the final product. Stripping is carried out at moderate vacuum and relatively low temperature, resulting in higher product yield and quality at reduced capital and operating cost.

The Evaporator/Stripper offers the following advantages:

Compared to packed columns

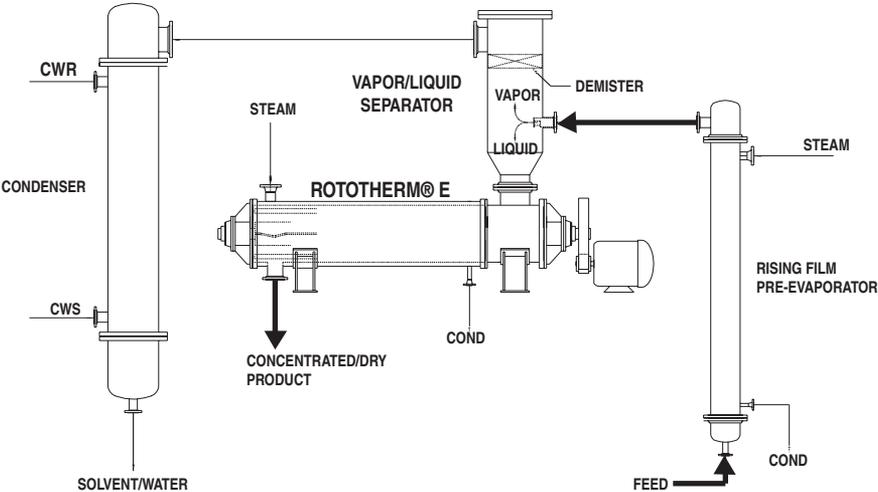
handle higher viscosity (5,000cP)
 lower pressure drop (0.05 Torr/stage)
 shorter residence time (<2 min.)
 handles solids, emulsions&suspensions
 less prone to fouling
 easier to clean

Compared to WFE's

no moving parts
 multi-staged
 single pass operation
 higher yield
 lower residual solvent
 lower capital&operating cost

Examples:

- Steam strip hexane from vegetable oil containing Lecithin from 80% to 2 PPM without product degradation.
- Remove Methyl Ester from crude Coconut oil from 50% to less than 1%.
- Dehydrate crude Vanillin from 20% to less than 50 PPM.
- Remove hexane from Marigold oleoresin from 10% to 25 PPM
- Strip cholesterol to less than 100 PPM from butter fat and lard.



Drying Strategies for Flavors and Food Ingredients

Problem: Producing free flowing powders with little or no moisture from liquid or slurry food products in a single pass.

Solution: Artisan's mechanically agitated thin film processors are widely used to produce free flowing powders from a variety of food ingredients. Rapid heat transfer, plug flow, and extremely short residence time permit concentration and drying of the most heat sensitive, foaming/fouling products from very dilute concentrations all the way to powder in one single positive pass through the Rototherm. In most applications, this eliminates one operating step; either a pre-concentration, or a drying step. In addition, significant capital and energy savings (factor of 2-3) are realized when the Rototherm replaces a spray dryer. However, If the final moisture specification requires spray drying, significant amount of energy is saved by feeding a more concentrated solution to the dryer, with the added benefit of preserving more of the valuable volatile flavor compounds.

Examples:

- Drying a heat sensitive protein containing fiber and starch by evaporating water from a 2% aqueous solution to a free flowing powder with a moisture content of less than 5%. This application eliminated a lyophilizing step.
- Concentrating and drying an aqueous soy isoflavones stream from 2% solids to a free flowing powder containing less than 1% moisture.
- Concentrating and drying aqueous niacinamide from 50% moisture to a dry free-flowing powder with no product degradation.

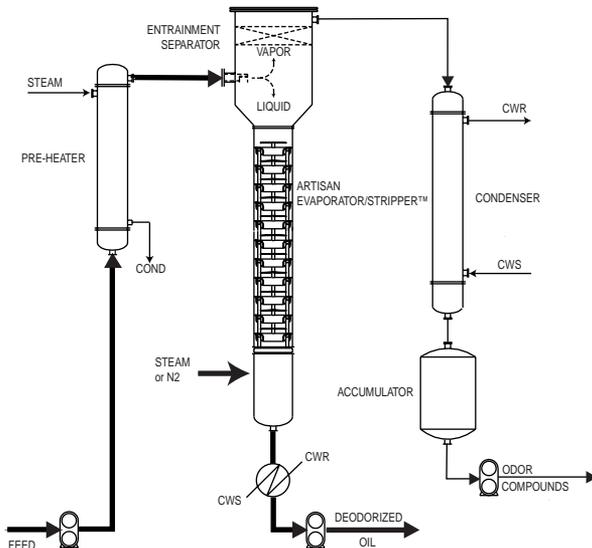


Deodorizing Edible Oils and Oleochemicals

Problem: Stripping unpleasant odors, such as free fatty acids and other odor causing volatile organic compounds from heat sensitive edible oils to non-detectable levels with no product degradation, or color formation.

Solution: Artisan proprietary falling film Evaporator/Strippers™ are widely used to strip the odor causing components from heat sensitive edible oils and other heat sensitive products. Relatively short residence time (60 to 120 seconds), high surface to volume ratio, and extremely low pressure drop make it the most economical alternative to vertical wiped film evaporators (WFE's). For stripping applications that are mass transfer controlled (i.e. diffusion limited) , one Artisan multi-staged Stripper, can replace two or more WFE'S.

Example: Deodorize cocoa butter by steam stripping the low molecular weight free fatty acids to non-detectable levels. Deodorization is accomplished at 10 torr, and relatively low temperatures (below 150°C) to prevent intra-esterification, while utilizing economically favorable stripping steam rate of 3%.



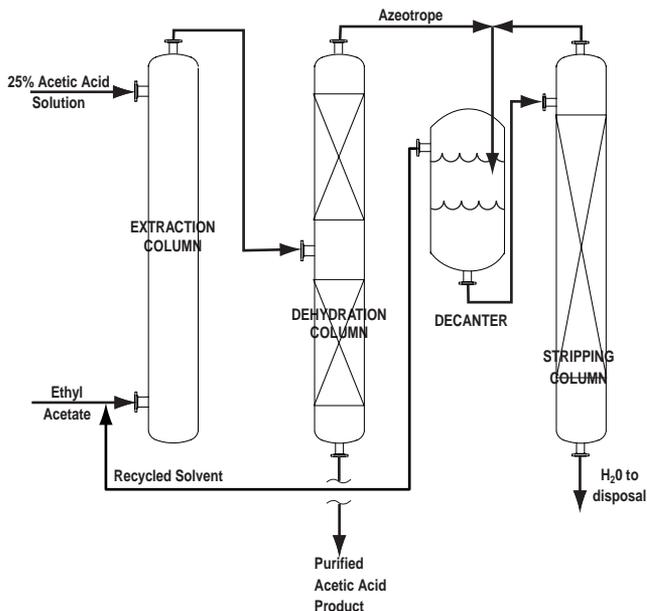
Recovering Selected Organics by Extraction

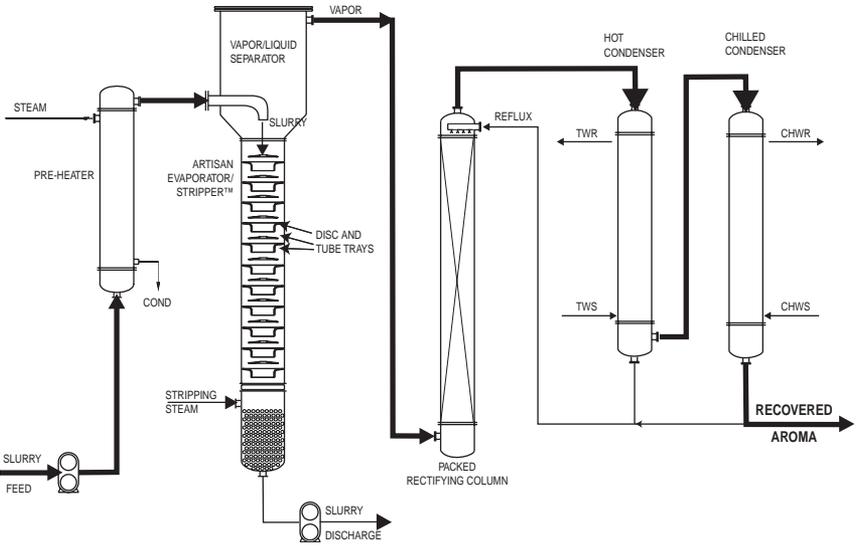
Problem: Recovering high purity organics from azeotropic or close boiling aqueous solutions is uneconomical using conventional distillation.

Solution: Use a closed-loop, cost effective, energy efficient Artisan controlled cycle liquid-liquid extraction system to extract the desired organic using a solvent with a higher affinity for the organic than water. Then use conventional distillation to separate the solvent from the organic. Purify the raffinate water in a steam stripping column to produce disposable water. Distillation overheads are recycled back to the recovery system, creating a process with no waste stream.

Example:

- Extracting theobromine and caffeine from an aqueous solution of chocolate liquor using methylene chloride as solvent. 98% of the theobromine is recovered in the Artisan Controlled Cycle Extraction column





Aroma Recovery

Problem: Recovering valuable flavors and aromas from beverages is typically accomplished by steam stripping under vacuum to preserve the volatile flavors. For most applications, such as in coffee or tea processing, it is more desirable to recover the flavor/aroma compounds directly from the raw material (slurry) as opposed to the extract (liquid). This will dramatically improve the yield and enables substitution of expensive flavor additives from processors own material.

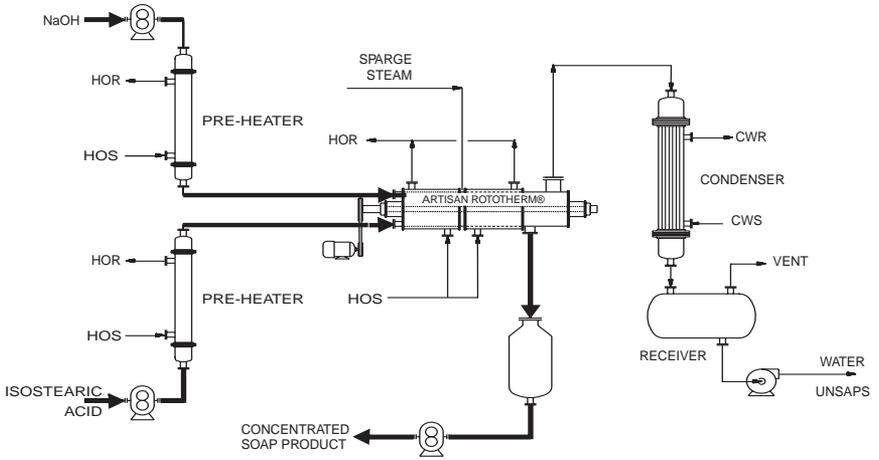
However, current technologies such as wiped film evaporators, and other mechanically driven rotary type devices have many disadvantages.

Solution: The Artisan Evaporator/Stripper® is the only “multi-staged non-mechanical” thin film processor capable of stripping valuable flavors/aromas directly from a slurry. The Evaporator/Stripper not only costs considerably less than the competing technologies to install and operate, it offers additional feature/benefits:

Feature	Benefit
• No moving parts	No maintenance
• Low residence time	No product degradation
• Multi-staged	Superior mass transfer
• Low gas/liquid ratio	Saves stripping steam
• Low pressure drop	Low pressure/temperature
• Ease of operation	Minimal supervision
• Easy to clean	CIP/aseptic processing

Examples: Recovering flavors/aromas from fruit and vegetable juices or purees, dairy products, alcoholic beverages, hot beverages such as coffee and tea, botanicals, herbs and spices, and citrus by-products.





Continuous Chemical Reaction with Product Purification

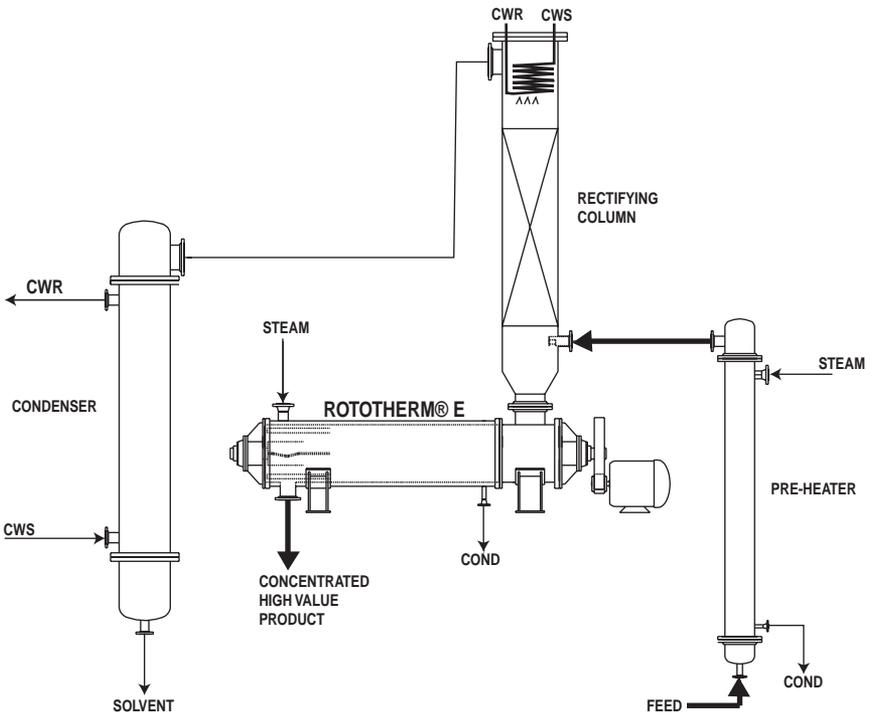
Problem: Equilibrium reactions, such as saponification and esterification are typically carried out in batch reactors, taking 24 to 30 hours to reach the desired conversion. Furthermore, it has been demonstrated that rapid removal of reaction by-products, such as water in esterification reactions, significantly reduces reaction time. However, both heat and mass transfer rates are limited in batch reactors. Consequently, as the reaction proceeds toward completion, water removal rate drops dramatically, inhibiting the reaction rate, and tying-up valuable reactor time.

Solution: The Artisan horizontal mechanically agitated thin film processor provides a unique combination of efficient heat and mass transfer capability, short residence time and mechanical integrity to perform continuous reactions of highly viscous, foaming, fouling and solids containing products. The rapid evaporation of reaction by-product(s) enables near complete conversion of most saponification and esterification reactions in minutes, yielding superior products, while minimizing energy consumption.

Examples:

- Single stage saponification of isostearic acid with sodium hydroxide, forming a stoichiometric soap, and steam stripping the “unsaps”, discharging a paste purified soap.
- Solvent-free saponification of sterol ester with sodium hydroxide, producing a heavy soap, followed by continuous recovery of free sterols from the soap.





Purifying Natural Flavors and Extracts

Problem: Purification of high value, heat sensitive, viscous, solids containing, foaming, and/or fouling aqueous and organic products poses tremendous challenges to the process engineer, especially when extremely low residual contaminant levels are required without the loss of valuable ingredients. This is particularly applicable to thermally sensitive materials, such as plant extracts, where exposure to process temperatures must be minimized to prevent product degradation.

Solution: Use the Artisan proprietary “mechanically agitated thin film evaporator” with a fractionating column to purify and concentrate your product. Strip the undesirable contaminants to non-detectable levels with no loss of valuable aroma/flavor compounds. The highly turbulent film, extremely short residence time and rapid surface renewal make the Rototherm® the most suitable evaporator/concentrator for your difficult purification problems.

Examples:

- Concentrate and purify nut flavor extract from a mixture containing dissolved solids (product), alcohol, water, propylene glycol and 20 PPM contaminant. The contaminant is reduced to 10 PPB, while concentrating the extract by six (6) fold.
- Purify orange peel oil by removing a variety of pesticides to less than a combined 1 PPM by evaporating D-Limonene in the Rototherm®.
- Concentrate citrus oil by evaporating D-Limonene in a Rototherm® with a fractionating column to concentrate Decanal, while minimizing product carry-over. Due to the short residence in the Rototherm, product color and quality is superior to those concentrated by batch distillation.

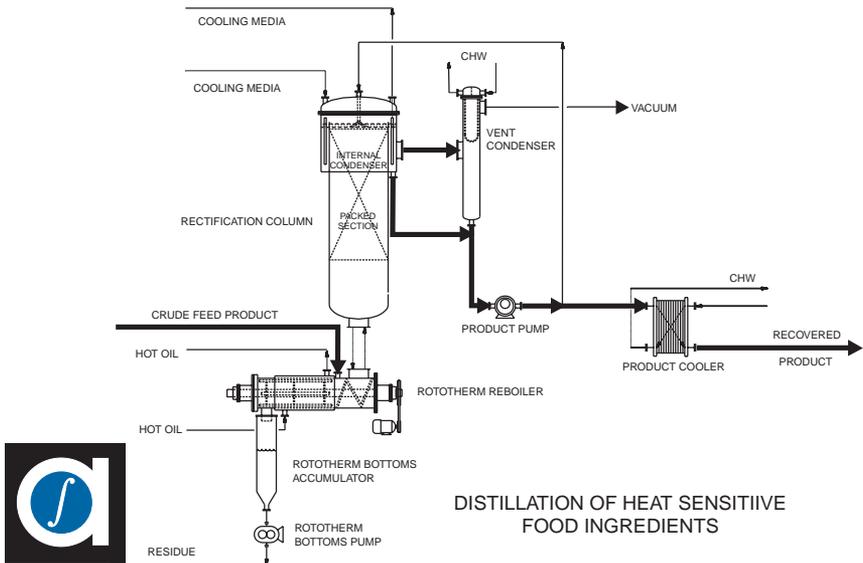


Distilling Heat Sensitive Food Ingredients

Problem: Distillation and purification of heat sensitive, viscous, or solids containing valuable food additives requires gentle heating and minimal exposure to time and temperature to prevent product degradation. This is nearly impossible to achieve by conventional technologies, such as thermosyphon, forced circulation, and even falling film reboilers.

Solution: Use Artisan's Rototherm® "thin film evaporators" as column reboilers for your most demanding distillation and purification applications. The highly turbulent film, combined with residence times ranging from 60 to 90 seconds, allows for nearly complete recovery of your valuable products without any damage to the appearance or functionality of the product. In addition, the Rototherm is capable of discharging the residue as a liquid, slurry, paste, or even solids, minimizing waste disposal costs.

Example: Purification of antioxidants used as preservatives in variety of products including, detergents/surfactants, oils, and food additives such as vitamins, flavors, and plant extracts.







65 years of chemical processing

73 Pond Street Waltham, MA 02451-4594

781-893-6800 fax: 781-647-0143

info@artisanind.com

www.artisanind.com