CONTINUOUS SOAP MAKING FROM NEUTRAL OILS

GENERAL PRINCIPLES

There are two main objectives in soap making from neutral oils or nearly neutral oils are as follows: -

- To produce economically the maximum quantity of neat soap at a constant and high TFM containing specified levels of electrolytes.
- The extract the economic optimum amount of glycerol

In some cases glycerol cannot be extracted economically and it will be appropriate to adopt an the Semi boiled/ SWING route of Soap making. Indeed right through 2002 till date Semiboiled has made more economic sense than traditional process in Africa and elsewhere except South and SE Asia. SE Asia has been mainly on DFA process due to its large installed capacity of DFA processes and associated oleo-chemical industry.

The recommended soap making plant consists of saponification, washing, fitting and separation stages and can include continuous spent lye neutralization notably when caustic soda is in fitting. The successful application of continuous spent lye neutralization however depends both on the configuration of the soap making plant and the operating strategy i.e. long period of steady state operation are required.

The following principles apply in the design and operation of continuous soap making plant: -

- The individual process steps in soap making are interdependent in that they are not only linked by the sequential passage of soap but also by the recycle of lyes
- Production runs should be maximized and frequent soap base changes avoided in order to meet the objectives specified above.
- If centrifugal separation is used, the capacity of the process is governed by the number of centrifuges available based on one centrifuge producing, 0.0 – 3.5 tons/hr of neat soap.
- Variability of the composition of input steams should be minimized. This requires accurate blending of oils as well as accurate dosing of caustic soda to saponification, brine and water to the washing unit, water at the fitting stage and caustic soda at the fitting stage if required.
- The hold up of product in each of the process stages should be minimized to aid process control and minimize downgrading losses during base changeovers.
- The addition of water to the system should be minimized, consistent with achieving product specification, in order to produce the highest concentration possible of glycerol in spent lye.

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

In this respect any water fed to the process should, in principle pass through the process counter-currently to the soap flow.

In practice this is not achievable as caustic soda solution is added to the process at the saponification stage and therefore to minimize dilution the caustic soda concentration should be as high as possible (47 – 50% W/W)

- The addition of caustic soda to the process should be minimized consistent with the complete reaction of the oils. When fitting with water, caustic soda enters the process only at the saponification stage. However, when fitting with caustic soda solution to produce a neat soap containing both sodium chloride and sodium hydroxide at the specified concentration then caustic soda enters the process at the saponification and fitting stages. In this case apart from the small amount of excess caustic soda which leaves the process at the specified concentration in neat soap, the residual excess is found in spent lyes and requires neutralization before glycerol evaporation.

In practice, it is not always possible to adhere strictly to the above principles and there is a need to consider carefully the implications of any deviations for the design, operation and economics of soap making.

SAPONIFICATION

Several saponification systems have been used in the soap industry. Batch saponification is still widely used and in certain circumstances (low tonnages, large number of bases) it may still be justified.
The disadvantages of batch saponification are: -

- It is labor-intensive involving skilled operators in what can be an unpleasant environment.

- It is energy intensive in terms of steam requirement.

Therefore, even in the cases described above, the decision to proceed with batch saponification plants in new installations requires careful and detailed economic evaluation, which is unlikely to prove positive. Consequently, batch saponification is not described further.

There are several types of continuous saponification system in operation within soap industry. The recommended system based on a mixer / tube reactor combination is described below. This system is seen to have advantages over all other systems in the following areas: -

- Accurate dosing systems: compatible with downstream continuous spent lye neutralization operations if practiced, and largely independent of pressure fluctuations in the reactor.

- Process intensive: reduces hold up in processing and gives space to utilization efficiency

- Opportunity to recover some of the heat of reaction for preheating wash liquor.

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Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

- Simple design, construction and operation.
- Excellent steam economy**:** oil preheating only during normal operation.
- Totally enclosed: improving the working environment and importantly, obviating the need to pump crude soap.
- Similar capital cost to other systems.

The recommended continuous saponification system consists of the following elements: -

- Oil and caustic soda dosing systems.
- Oil heater.
- In – line mixer for the production a reactive emulsion of oil and caustic soda.
- Pressurized reaction vessel (tube reactor).
- System for cooling the crude soap product with spent lye before transfer to the lye neutralization / washing stage of the process.

It should be stressed that the element of the saponification plant described in more detail below form part of an integrated reactor system and deviation from the recommended design in one area e.g. dosing system may have undesirable consequences.

OIL AND CAUSTIC SODA DOSING SYSTEM

The accurate dosing of oil and caustic soda to the saponification reactor independent of any pressure fluctuations inside the tube reactor is a key requirement in the soap making process. Caustic soda in excess of the stoichiometric requirement of the oils to be saponified will emerge from the process as spent lye and will require neutralization before downstream glycerol evaporation. Irrespective of the method of neutralization, there will be a cost associated with this neutralization operation. It is therefore highly desirable to produce spent lyes with the minimum concentration of caustic soda consistent with complete saponification of the oils. Conversely, it is important that adequate caustic soda is metered to the process such that there is no risk of unsaponified oil emerging from the process in neat soap. The recommended system to achieve the desired dosing accuracy is to use a multihead volumetric piston pump equipped with an independent flow check or flow control system. In some locations particularly where volumetric piston pumps are not manufactured locally and the cost of import is prohibitively expensive, then it may be necessary to consider alternatives. The key principle in the selection of alternatives is to choose a pump where the delivery is largely unaffected by pressure fluctuations in the reactor and therefore positive displacement pumps are preferred. Process Engineers should be consulted before alternatives are selected for advice in this area. Independent positive displacement pumps on each stream with flow control using VVF drives without altering the stroke lengths have also proven to be very reliable in the field.

The dosing pump should have 3 dosing pairs for each component (oil and caustic soda) driven by single motor. It must be possible to isolate each head independently for repair if required. This configuration gives the following advantages:-

- Simple throughput changes maintaining the oil: caustic soda ratio by manipulation of the variable speed drive.

- Simple, accurate oil: caustic soda ratio changes by manipulation of stroke lengths.

- Minimization of pulsations by the use of 3 heads per component set at 120^{o} out of phase, removing the need to fit pulsation dampeners which can be unreliable.

The dosing pump should be protected by filters and pressure relief valves. Particular attention must be given to the design stage to ensure a safe discharge from these valves. As a further precaution, it is recommended that the pump drive be interlocked with over-pressure protection switches in both the oil and caustic soda delivery lines.

Pressure sustaining valves are required in both delivery lines to ensure that materials from the feed tanks cannot simply "run through" the pump at start – up before a back pressure is generated in a reaction vessel.

The degree of sophistication of the flow check / control system for the pump will vary from application to application. Alternatives, which may be considered, are as follows: -

- **Coriolis type mass flowmeter**: this type of flowmeter allows the simultaneous measurement of density, which in turn could be used as part of a sophisticated control system to compensate automatically for changes in caustic soda concentration. Experience has shown that manufacturers' guidelines for installation must be strictly adhered to for the successful use of this type of flowmeter. E.g. installing it in vertical bends to ensure no deposition in the measuring element for better accuracy

- Magnetic flowmeter: this type of flowmeter is suitable ***caustic soda only*** to give a volumetric flowrate indication. It may be combined with a separate density meter to perform the same function as coriolis type mass flowmeter.

- Variable area flowmeter: this type of flowmeter can be used for both oils and caustic soda. It is the least accurate of types discussed.

In some cases, and particularly where exceptional accuracy is required, then the flowmeter can be incorporated into a flow control system where the total flowrate of both components and the ratio between components can be controlled. Automatic stroke length adjustment on at least one head of the pump would be required i.e. manual coarse adjustment, automatic fine adjustment.

The dosing pump should be fed from constant level tanks mounted above the level of the pump to ensure a flooded pump suction feed. The preferred method to ensure a constant level in the feed tanks is to provide an overflow system. If the main storage tanks are located adjacent to the head tanks then these tanks can simply overflow back to the storage tanks. This option is particularly attractive and is recommended for greenfield installations. If this is not the case then a separate overflow tank is recommended. In this case the overflow tank will receive a constant flow from the head tank and will discharge via a centrifugal pump back to the main storage tanks. Either system gives the following advantages: -

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Level 4, Dynasty, "A Wing"
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

- Flows of materials are continuous thus avoiding the risks of solidification in transfer lines.

- Head tanks and overflow tanks can be emptied completely for maintenance or other purposes.

- The level in the feed tanks is constant thereby ensuring consistency of feed to the dosing pump improving its dosing accuracy.

Appropriate alarms are required in the feed tanks as in an emergency actuated shut – off valve on the caustic tank. Each tank is equipped with lines to recycle the feedstock from the dosing pump back to the tank in order to stabilize flowrates before start – up.

OIL HEATER

The recommended oil heater is a shell and tube exchanger with stainless steel tubes through which the oil passes. Shell and tube designs have proven to have lower maintenance requirements for this duty than plate types. The heat exchanger must be capable of heating the oil to 105^{o}C. A mixture of oil at 105^{o}C and unheated caustic soda will produce an emulsion temperature of approximately 80^{o}C. It is unlikely that a higher emulsion temperature will be required although it is sometimes necessary for emulsion stability reasons to use a lower temperature where the oil may need to be heated only at 80^{o}C. This is particularly relevant when the caustic soda supply contains high levels of salt. This factor must be considered when sizing the flow control valve for steam to the heat exchanger so that it will operate in a suitable manner over the desired range.

IN – LINE MIXERS

The function of the mixer is to create a reactive emulsion of caustic soda in oil. The finer the emulsion, the more rapid the initial rate of saponification. Other factors, which affect the reaction rate, are as follows: -

- The higher the concentration of free fatty acids in the oil blends, the quicker the initial reaction rate.

- The recycle of soap to the mixer will increase the overall reaction rate but will also increase the power consumption of the mixer. This method can only be applied when dynamic mixers are used.

- The higher the emulsion temperature, the quicker the reaction rate. As noted earlier in "**Oil heater"** there is sometimes a balance to be struck between increasing the reaction temperature to increase the rate of reaction and decreasing the reaction temperature to aid emulsion stability.

Two types of mixers can be considered as part of the recommended saponification system: -

- Dynamic mixers.

- Static mixers.

Address : Innoleague
Level 4, Dynasty, "A Wing"
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

DYNAMIC MIXERS.

Three types of dynamic mixer have been used extensively in the soap industry

- Ystral type
- Supraton type
- Cavitron type

Cavitron mixers are favoured but the final choice will depend on various factors such as local service centre, design improvements etc. Process Engineers should be consulted to advice on choice.

Dynamic mixers function by applying shear to the feed stocks, which are fed into the centre of the mixer via concentric tubes and are forced to pass through intense shearing zones in their passage to the periphery of the mixer where the emulsion thus formed is discharged. The efficiency of mixing can be checked by taking a sample of the emulsion, which should boil and foam over the receptacles on standing for less than 1 minute. Extreme caution is required when performing this check and same precautions as used when handling caustic soda should be applied.

Saponification is an autocatalytic reaction and is normally described as consisting of 3 stages:
-

- An induction period (up to 20 – 30 % conversion)
- A steady state period (up to 80 – 90 % conversion)
- A slow down period as reactants are consumed.

The use of high shear dynamic mixer virtually eliminates the induction period leading to shorter reaction times. Other advantages for dynamic mixers are: -

- Consistent emulsification virtually unaffected by flowrate.
- Less risk of blockage in the mixer when the oil: caustic soda ratio is destabilized for any reason
- Proven ability to operate with excess oils when continuous spent lye neutralization is practiced.

The free fatty acid concentration in oils can have a marked effect on reaction rates. Although dynamic mixers can still produce highly reactive emulsions when the free fatty acid level is below 3%, when the concentration is below 2% then difficulties can be encountered. Normally this effect can be countered by increasing the residence time for reaction (operation at below design throughput) and / or manipulation of the emulsion temperature. Companies who receive oils with free fatty acid contents below 2% are advised to consult Process Engineers for advice on operation and reactor design.

The disadvantages of dynamic mixers are: -

- High capital cost
- Energy consumption (up to 6 Kw/ton of emulsion)
- Pressure flushed double mechanical seal requirements

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

- Pumping action.

Cavitron and Supraton dynamic mixers can be compared with three stage centrifugal pumps in their pumping action. Consequently and particularly at start–up, there is a tendency for the mixer to draw feedstock through the dosing system if the latter is not correctly designed. Companies intending to purchase dynamic mixers are advised to consult Process Engineers for up- to- date information on developments aimed at reducing the disadvantages of these devices.

STATIC MIXERS

In some cases the choice of dynamic mixer may not be appropriate and a static mixer may be selected as an alternative. This is normally the case when skills for maintenance and for overhauling the machines are unavailable. The type of mixer recommended in these circumstances is the margarine mixer. The margarine mixer uses the fluid energy to provide elongation, which is adequate to form a reactive emulsion. In contrast to dynamic mixers the induction period of the saponification reaction is significant and consequently reaction times are longer which must be taken into account in the design of the subsequent reaction vessel.

Advantages of Margarine mixer are: -

- ***Simple construction***: local fabrication possibilities
- Low cost
- No maintenance requirements except for occasional corrosion checks.
- Minimal energy consumption / pressure drop
- Simple connection / dismantling for cleaning.

The disadvantages of this type of mixer are: -

- The turn down ratio has never been established. Consequently the units are designed for a fixed throughput (± 10%)
- The effect of operating with excess oil on the overall saponification reaction, as is required during continuous spent lye neutralization, has not been established.
- Care is required at star – up to ensure that the mixer does not block due to feedstock flowrate instability.
- Longer overall reaction times in comparison with dynamic mixers resulting in a need for a longer residence time reaction vessel.

They are not recommended for use with oils with low free fatty acids (below 3%) as the induction period of the reaction will become excessively long.

PRESSURISED REACTION VESSEL / TUBE REACTOR

A reaction vessel is required after the mixer to provide residence time for the reaction to complete. The recommended residence times are as follows:

- 12 minutes when used in conjunction with an approved dynamic mixer.

- 20 minutes when used in conjunction with the margarine mixer

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

- 25 minutes when used in conjunction with an approved dynamic mixer for Semi boiled/ SWING. If this is not available especially when the same plant has been converted over to SWING from an earlier design then an additional residence may be given by incorporating a receiving tank with a recycle loop.

For conventional saponification the extra cost of providing a reaction vessel with a residence time of 20 minutes even when used in combination with a dynamic mixer should be established when considering installation of such a system. In some cases the extra cost will be justified by future flexibility to increase throughput or to handle low FFA oils.

The vessel has several essential design features: -

- It is a tube reactor without internal elements designed to pressure vessel codes. The designed pressure is 6 bar at 150^{o}C and care must be exercised in the connection of steam line to connecting pipe work to ensure that the design pressure is not exceeded.

- The diameter and height of the vessel are determined by the residence time allowing for a cross sectional area of 0.09m^{2}/ton/hr of oil. This figure may change with further experience and Process Engineers should be consulted for up – to – date recommendations.

- The vessel consists of a tubular section with two end cones. The cones should be flange fitted to the tubular section to allow dismantling for internal inspection

- The vessel is equipped with a steam coil to maintain the temperature of the soap in the vessel in the event of a temporary production stoppage. Soap may be left inside the vessel (but not inside the mixer) for up to 24 hours.

- A pressure of 2 – 3 bar is maintained in the vessel during normal operation by the action of a pressure control loop acting on a half ball type valve. These valves allow virtually full bore pipeline flow in the event of partial blockage considerably easing problems of this nature. The vessel operates under pressure to avoid boiling inside the reactor, which can result in a wide residence time distribution and a reduction in the reaction temperature. Both effects are detrimental to the achievement of full conversion inside the reactor.

- The vessel should be constructed from a hot rolled nickel / steel laminate. Process Engineers should be consulted before other materials of construction are considered.

- The vessel should be protected by a bursting disc mounted in the pipe work adjacent to the reactor outlet. The safe discharge of a product from the bursting disc in the event of failure is essential and connecting pipe work to an expansion vessel should be self-draining.

- Sample points are required on the inlet and outlet pipe work from the reactor to enable checks of emulsion reactivity and conversion.

The reactive emulsion formed by the mixer at approximately 80^{o}C is transformed into crude soap at a temperature of 130 – 140^{o}C at approximately 72% TFM at the outlet of the reactor.

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

The soap needs to be cooled before passing through the washing unit from a temperature of 130 – 140°C to 90 – 96°C to prevent boiling, and diluted from 72% TFM to approximately 36% TFM to reduce its viscosity. Therefore to achieve both cooling and dilution, lye from the washing unit is recycled into the crude soap stream.

LYE RECYCLE

Lye is fed from the washing unit via a buffer tank to a two – stage cooler where the temperature is reduced to 60 – 70ºC before being added to the crude soap stream immediately downstream of the pressure control valve of the reactor.

The lye buffer tank is required for the following reasons: -

- It provides a reservoir of lye for start – up.
- It is partitioned for lyes originating from different bases (if required)
- It decouples the washing unit from the reactor. Washing units operate most efficiently when the flows in and out are steady and constant. The incorporation of the lye buffer tank allows the flow of lye from the washing unit to be independent of the "demand" for lye recycle.

The lye cooler consists of two stages: the first stage allows some recovery of heat from the lye into the diluted brine component of wash liquor while the second stage allows for cooling of the lye to the target temperature using water as cooling medium. Essential design features include: -

- A by – pass for the dilute brine to the first stage exchanger to avoid overcooling the lye if the lye temperature is low e.g. after prolonged shutdown.
- A system to clean the second stage heat exchanger. Over a period of time the second stage heat exchanger fouls at the prevailing operating temperature with soap, which precipitates from solution. It should be cleaned by flushing for a few minutes with low pressure steam via the cooling water circuit while simultaneously circulating dilute brine solution counter – currently through the lye circuit at 5 x the normal flowrate. Frequently dismantling for cleaning is not recommended.

The second stage heat exchanger should be oversized by 25% to allow sufficiently long periods of operation between cleaning.

The recycle of lye not only cools and dilutes the crude soap but also aids in the glycerol extraction process. Firstly, some of the water in lye will be absorbed into the crude soap increasing the concentration of glycerol in the spent lye. Secondly, a "washing" process is performed by mixing and subsequently allowing separation. Consequently, it is essential that the crude soap and recycled lye are well mixed.

SOAPWASHING AND IN SITU SPENT LYE NEUTRALISATION

Soap washing is required as part of the soapmaking process. Glycerol is extracted from soap to produce spent lye using a mixture of diluted brine and nigre lye as wash liquor.

Address : Innoleague
Level 4, Dynasty, "A Wing"
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

When designing glycerol extraction equipment, there is an economic balance between the value of glycerol in spent lye and the capital cost of the equipment. Other factors notably operating strategy and the availability of local skills also require consideration. The existing installations, techniques are available to estimate the economic optimum yield of glycerol in soap washing. These techniques seek to optimize the glycerol content of spent lye and that in washed soap by manipulation of the wash liquor: soap ratio.

In addition to the extraction of glycerol at the soap washing stage, it is also possible to practice in situ spent lye neutralization. This process reacts residual oil present in the soap curd from the saponification stage with excess caustic soda originating in fitting when it is necessary to use caustic soda for fitting to produce neat soap containing both sodium chloride and sodium hydroxide. In this case caustic soda enters the soapmaking process at two points:

- In saponification
- In fitting

Excess caustic soda, other than that which is present in neat soap after the separation stage of the process emerges in spent lye. Although it is possible to avoid the addition of excess caustic soda in saponification, it is inevitable that some of the caustic soda added at the fitting stage will be present in the nigre lye, which separates from neat soap. Nigre lye is subsequently recycled to the washing unit as a component of wash liquor and the caustic soda in nigre lye will ultimately emerge from the process in spent lye. The presence of caustic soda in spent lye is wasteful because it is an unutilized raw material and hydrochloric acid is required for its neutralization in lye treatment. It should be stressed that the concentration of caustic soda in spent lye should not be allowed to drift to zero as this signifies that there is insufficient caustic soda in the overall system to satisfy the stoichiometric requirements of the oils.

In situ lye neutralization takes place in washing units concurrently with glycerol extraction using residual oil present in the soap feed resulting from the addition of less than the stoichiometric quantity of caustic soda to the saponification reactor.

For the successful application of in situ lye neutralization, the following factors are of importance:

- The dosing system for oil and caustic soda to the reactor must be highly accurate such that the ratio between the flowrates of the components is accurate to 0.2% i.e. approximately, 0.8Kg of caustic soda in excess of deficit from set point per ton of oil.
- The saponification reactor needs to be tolerant of the higher viscosity soap curd containing free fat. This requirement is met by the dynamic mixer / tube reactor combination.
- The flowrates and combinations of feed and discharge streams from the washing unit should be constant. This implies a constant hold-up of soap in the washing unit, which in turn implies a constant proportion of free oil in the soap available to react with excess caustic soda in wash liquor. In practice, constant hold-up is more difficult to achieve in rotating disc contactors than divided pan units. The other significant implication arising from this condition is that the composition and flowrate of nigre lye

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

should be constant. This can be difficult to achieve in systems where pan separations is utilized. In plants where there are frequent base changes i.e. more than once per day or where the throughput of the plant is varied to match downstream finishing capacity, then achieving the target caustic soda concentration of 0.2% in spent lye will be impossible and higher target of 0.5% should be set. It should be noted that even partial neutralization will give economic advantages.

The recommended procedure for the implementation of the in-situ lye neutralization is as follows:

- Start up the saponification reactor using approximately 1.05 x stoichiometric requirement of caustic soda and continue to produce soap until the whole soapmaking process has reached steady state.

- Reduce the caustic soda flowrate to the reactor in 2 – 3 steps monitoring the caustic soda concentration in spent lye. During this period, the washing unit should be carefully monitored to check if the soap becomes undergained due to the loss of electrolytes. This would need to be compensated for by raising the salt concentration in wash liquor.

During in situ lye neutralization a minor loss in washing efficiency will occur as glycerol is "liberated" from oil during its passage through the washing unit rather than being available for extraction on entry to the washing unit. This loss in efficiency is small and can be compensated for by a small increase in the wash liquor: soap ratio although such action is not generally required.

Two types of continuous counter – current washing units are in common use in soapmaking for glycerol extraction and in situ lye neutralization if required: -

- The Rotating Disc Contactor (RDC)

- The Divided Pan Unit (DPU)

The choice between an RDC or DPU for the soap washing duty depends on several factors including the following: -

- *Capital cost*: local fabrication opportunities for DPUs

- *Operational flexibility*: DPUs are preferred when frequent changes of throughput or soap base are necessary because interface control is difficult with RDCs under change – over conditions.

- *Operating skills:* DPU are considered easier to operate than RDCs and hence lower free caustic soda targets in spent lye can be applied when in situ lye neutralization is practiced.

- *Extraction Efficiency*: RDCs are capable of producing higher glycerol concentrations in spent lye than DPUs at the same wash liquor: soap ratio and residual glycerol in washed soap.

- RDCs can be sited outside buildings giving some economy in space requirements. The choice between RDCs and DPUs for the soap washing duty is complex and

Address : Innoleague
Level 4, Dynasty, "A Wing"
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

Process Engineers should be consulted to asses each case individually before a decision is made.

ROTATING DISC CONTACTOR (RDC)

The Rotating Disc Contactor (RDC) is a mechanically agitated counter – current extractor. It is a vertical cylinder consisting of compartments separated by stator rings fixed to the shell. In each compartment, a rotating disc is mounted on a vertical shaft. Both ends of the cylinder are expanded to provide settling chambers for the two phases. The vessel is hot water jacketed and constructed from AISI 316 stainless steel. Diluted soap from saponification enters just below the bottom compartment but above the bottom-settling chamber.

The wash liquor enters between the top compartment and the top-settling chamber. The wash liquor is heavier than soap and drops down the column while the soap rises and both phases will be mixed while passing through each compartment.

In bottom settling chamber, an interface is present between spent lye and the soap / spent lye mixture. The interface position can be detected by the measurement of differential pressure over the settling chamber and the signal from this sensor is used to control the rate of off-take of spent lye. The parameters in the control algorithm should be adjusted such that the spent lye flowrate is as constant as possible. On / off control which can occur when the control parameters are poorly adjusted results in hold – up variation inside the RDC leading to poor washing efficiency and surges in the flow of soap from the RDC

Other key factors affecting the design selection and operation of RDCs for soap washings are as follows: -

- The flows of all streams into and from the RDC should be constant in terms of flowrate and composition. The hold-up of soap in the RDC depends on the flow and composition of the soap and lye i.e. dilute wash and wash liquor into the unit and spent lye out. Hold-up variation causes variation in the flowrate of composition of washed soap, which can be transmitted to the fitting and separation stages of the process ultimately affecting the composition and quality of neat soap. Therefore the operating strategy for soapmaking plant containing an RDC should be to avoid more than one base change in 24 hours and to operate at a fixed throughput.

- Sight glasses must be provided to ensure that the operator can check the interface position in the lower settling chamber.

- The rotor operates at a speed fixed according to the throughput and RDC geometry. RDC designs are available for 8 and 15 tons/hr of wash soap at 54% TFM (i.e. oil flowrate of approximately 4.5 – 8.5 tons/hr). The maximum recommended turndown ratio of RDC is 60% and capacities of 110% of design can be used. Therefore with two designs available, washed soap flowrates of 4.8 – 16.5 tons/hr can be accommodated. The use of RDCs for lower capacities than 4.8 tons/hr of washed soap is not recommended.

DIVIDED PAN UNIT (DPU)

The DPU is a multi chamber counter – current mixer settler, originally made by dividing a soap pan into sections. Modern cascade DPUs rely on hydrostatic head balance principles for their operations. Soap is transferred from compartment to compartment using the kinetic energy of a flow of lye from a centrifugal pump, forcing the soap down an overflow weir to emerge at the bottom of the next downstream chamber. The soap, by virtue of its lower density, floats through lye present in the chamber to the surface next chamber and so on. Lye passes from chamber to chamber by an overflow mechanism counter – current to the soap flow. By – passing and channeling of the streams is prevented by the use of spreader plate (lye) and baffles (soap).

DPU designs are available for 5 and 10 tons/hr of washed soaps. Turndown ratios are similar to those of RDCs.

DPUs can be constructed as double units with one set of lye transfer pumps. This allows two soap bases to be processed sequentially fed by the same saponification reactor with minimum cross contamination of the soap bases at changeover.

FACTORS AFFECTING THE WASHING UNIT OPERATION

One of the key factors in the smooth operation of washing unit in general, but RDCs in particular, is the correct control of the wash liquor electrolyte concentration. The recommended method for continuous soapmaking plant is to control the density of the total wash liquor, i.e. the combined fresh diluted brine and nigre lye returning from the centrifuges such that the soap which emerges from the washing unit is very lightly “grained”. This will ensure that the soap within the washing unit is in the required region of the phase diagram for optimum washing. The density of the wash liquor is dependent upon temperature and electrolyte concentration. In addition, the type of electrolyte i.e. sodium chloride or caustic soda has different effects on density. However, because caustic soda is approximately 1.15 times more effective at salting out soap from solution (graining efficiency) but has a lower contribution to the wash liquor density than sodium chloride, it is possible to use density measurements to control the grain of the soap i.e. density is not being used to infer a particular electrolyte concentration but rather as a direct means of controlling then graining of the soap. Details of the equipment options available for the measurement of density are available. Although the technique described above for the control of the condition of the soap during washing above represent a considerable simplification over methods previously applied, problems can occur particularly if excessive quantities of nigres are re-introduced into the system for storage.

The nigres, which return to the washing unit from the centrifuges, will be in equilibrium with the soap from which it has separated.

In addition, at steady state, the flowrate of nigres will be constant and about 30% of the total wash liquor flowrate. However, it is recommended that a nigre buffer tank and flow control system will be utilized to remove flowrate fluctuations which will lead to instability in the washing unit. The composition of nigre les from storage can be variable and some form of segregation into those arising from different bases should be practiced. To prevent poor odour and colour and high glycerine in neat soap, nigre from storage should not be added to the wash liquor stream at > 40% of the total flowrate. The flowrate of fresh brine must be reduced correspondingly to maintain the correct and desired wash liquor: soap ratio.

Address : Innoleague
Level 4, Dynasty, “A Wing”
Andheri Kurla Road Mumbai 400069
Fax : +91 22 30500233
Email : sales.innoleague@gmail.com
Website : www.innoleague.com

FITTING AND SEPARATION

The objective of fitting and subsequent separation is to produce a neat soap with the following characteristics: -

- A high and constant TFM content
- A low dirt content
- Specified and constant electrolyte levels.

The achievement of the desired neat soap characteristics through fitting and separation depend on equilibrium soap phase criteria. The regions of interest in the phase diagram for both washing and fitting are G and H. Region H is a two phase region containing neat soap and essentially soap free lye.

Soap, which emerges from the washing system, should have a composition just inside region H at the interface between regions G and H. If left to settle, the washed soap at approximately 54% TFM would separate into neat soap and lye. In this case the electrolyte content of the neat soap would be entirely sodium chloride. Generally, it is common practice to operate the washing unit using wash liquor containing more electrolyte (sodium chloride) than is strictly required to preserve the two-phase system (overgrain). This practice ensures that the soap in the washing unit does not become undergrained i.e. operation in region G of the phase diagram, which would result in dissolution of soap in spent lye. Undergrain may occur in the washing unit due to changes in the soap base composition or in the wash liquor concentration, which may go undetected. To compensate for overgrain in the soap which emerges from the washing unit which in turn would result in neat soap containing than the specified level of sodium chloride, facilities must be available to inject water into the washed soap stream i.e. fitting with water, before separation. This process dilutes the sodium chloride content of the washed soap, which yields a neat soap with a correspondingly reduced sodium chloride concentration.

As an alternative, if for product performance/characteristics reasons the content of sodium chloride in neat soap is unacceptably high by fitting with water alone, then fitting with caustic soda alone can be used. Firstly water is added to the washed soap. This would change the composition of soap so that it would be represented by a point within region G of the phase diagram. Neat soap would be produced in this case at lower electrolyte content but a large proportion of soap will remain in the nigre, which separates from the neat soap, and reprocessing would be required. To adjust the composition of the soap such that it again could be represented by a point in region H of phase diagram and therefore yield only neat soap and lye on separation, electrolyte in the form of caustic soda is added. Although in this case the total electrolyte in the neat soap product is similar to that which it would have been if washed soap was fitted with water alone. Fitting with caustic soda solution allows both compositional corrections to be made and also allows sodium chloride substitution with sodium hydroxide. Sodium hydroxide can be neutralized at later stage in the process by fatty acids increasing the TFM of the soap and reducing the total electrolyte content of the soap to concentrations which would not be possible to achieve by fitting with water and separation alone. Where possible, fitting with water only should be practiced and is the best recommended procedure in normal circumstances given advantages of process simplicity and avoiding the need for in situ spent lye neutralization. However, care must be taken to ensure effective control of the process as the presence of excessive concentrations of sodium

chloride in neat soap, and ultimately in finished soap, can lead to bar cracking problems. In addition the need for accurate dosing of oil and caustic soda, the later at slightly above the stoichiometric requirement is emphasized as when fitting with water alone there is no "reservoir" of excess caustic soda to react with unreacted oil in the washing unit if for some reason, insufficient caustic soda is fed to the reactor.

The development of the modern soapmaking process has reduced the requirement of fitting to remove dirt from the soap. Where bleaching of the oils for soapmaking is practiced together with effective filtration and polishing filtration, then much of the residual colour in the oils is removed in the washing section of the process. If however, it is necessary to remove color during fitting and separation, then fitting must be carried out such that the soap composition is represented by a point within region G of the phase diagram. The nigre lye produced will absorb much of the color in the soap. This technique is more difficult to accommodate in continuous fitting and separation systems as the nigres are returned directly to the washing unit. In a batch separation process the nigres, after treatment to recover their soap content, can be discarded or downgraded into hard soaps taking with them the colour removed from the soap during the fitting process.

Although batch fitting and separation is still widely used in the soap industry, for new installations batch fitting and separation is unlikely to be selected for the following reasons: -

- The process consumes 0.2 – 0.4 tons steam per ton of 63% TFM soap.
- The pans in which separation takes place cannot be filled "brim full" and therefore the process requires more pans to meet the required capacity.
- Modern upstream processes have reduced the requirement to remove colour bodies during fitting and separation.

Consequently, batch fitting and separation is not described further. The recommended system for fitting and separation is continuous in–line fitting followed by centrifugal separation. If centrifugal separation cannot be justified because of the capital cost associated with replacing existing pans, local skill levels are not considered adequate to maintain the machines effectively or there is inadequate capacity or base fragmentation. Then in – line fitting coupled with batch settling is recommended.

IN – LINE FITTING CENTRIFUGAL SEPARATION

Soap at approximately 54% TFM is received from the washing unit into a buffer tank. It is important that the soap is pumped away from this tank at constant flowrate, which will be approximately 1.75 times the flowrate of oil into the process. The flowrate of washed soap from this tank should only be adjusted infrequently in response to high or low level alarms in the buffer vessel, thus allowing a constant and predetermined feed to the centrifuges. In turn, the flowrate of oil to the process should be fixed such that the correct and whole number of centrifuges are required for the separation i.e.: -

- 1 centrifuge available - oil flowrate 2 – 2.5 ton/hr
- 2 centrifuge available - oil flowrate 4 – 5 ton/hr
- 3 centrifuge available - oil flowrate 6 – 7.5 ton/hr

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- 4 centrifuge available - oil flowrate 8 – 10 ton/hr

Generally, centrifuges can be underfed without affecting their separation efficiency but overfeeding will result in poorer separation reducing the neat soap TFM and increasing the electrolyte content. (Hence where-ever possible feed centrifuges by positive displacement pumps)

Fitting liquors (water and caustic soda if required) are pumped into the flow of washed soap at a rate controlled by the washed soap flowrate such that the ratio between the fitting liquors and washed soap flowrates is kept constant. When fitting with water only, hot water (50^{o}C maximum) must be used to avoid the formation of middle soap. The fitted soap is circulated in a ring main at a recycle ratio of approximately 5:1 to mix the soap with fitting liquor and to feed each centrifuge connected to the ring main with soap at a fixed composition, temperature and pressure.

The separated streams of neat soap and nigre which emerge from the centrifuges are fed into buffer tanks or storage tanks directly if such tanks are local to the centrifuges and preferably are at a lower level than the centrifuges. The recommended centrifuge is a SOGW 600 manufactured by Veronesi. This unit is generally less expensive than other similar types on the market(e.g Alfa Laval). However there are constant changes in the market place hence exact recommendation should be obtained by your technology provider. Care however must be taken during installation to ensure that the configuration of the discharge piped does not result in excessive back pressure (.0.5 bar g) on the centrifuges or they may overflow and considerable damage can result. Key features concerning the design specification of the centrifuge are: -

- The centrifuge must be constructed from the Process Engineering recommended materials of construction as detailed in the Equipment Specification sheets.

- Ideally the centrifuge should be fitted with a permanent vibration transducer, which will not only act to stop the centrifuge due to excessive vibration but can also be used to indicate that cleaning is required. A vibration switch to stop the centrifuge in the even of excessive vibration is an acceptable alternative safety device. The vibration switch should not be used to automatically stop the feed to an individual centrifuge or overloading of other centrifuges connected to the ring main could result. If centrifuge is however stopped automatically due to excessive vibration the operator must be warned by an alarm system to take prompt action to isolate the centrifuge and reduce the washed soap flowrate to the fitting and separation section of the process commensurate with the number of centrifuges remaining in service. Failure to isolate the centrifuge would result in soap overflowing the unit.

- The centrifuge manufacturer will supply speed indication, pressure control valves and pressure gauges on each outlet. Sight glasses are required on each outlet and they should be checked regularly for wear and replaced as necessary.

- The feed line to the centrifuge should contain a feed point for hot wash liquor to allow flushing, a flow meter and a flow control valve such that the flowrate to each centrifuge operating in parallel can be balanced. While setting the flowrate of soap to the centrifuge it is important that the operator can see the ring main pressure gauge such that he is able to set the flows to each centrifuge and yet maintain the

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loop pressure at approx 0.5 bar g. It is important that the washed soap feed pump, the fitting liquor pump and the fitted soap recycle pump be fitted with over – pressure cut – out switches and mechanical pressure relief system.

- The drive system for the centrifuge must allow a gradual speed increase. Currently both inverter drive system and mechanical clutch systems are in use. Hydraulic clutches should not be specified. Process Engineers should be consulted for the latest recommendations on centrifuge drives.

Extreme caution must be exercised when purchasing and operating centrifuges. They are potentially hazardous machines. No deviation from recommended materials of construction should be specified to suppliers without consulting Process Engineers. Similarly all moving parts should be checked on a regular basis for stress cracking or other forms of corrosion. Records should be kept of the machine vibration and repairs or refurbishment effected promptly using ***only*** original manufacturer supplied parts.

IN – LINE FITTING AND BATCH SEPARATION

The principles of in – line fitting whether followed by batch separation or centrifugal separation are identical as are the main items of equipment. However, in the case of batch separation, the fitted soap is fed via a static mixer into a soap pan in which separation occurs rather than into a ring main and centrifuges.

In centrifugal separation it is a relatively straightforward task to control the fitting process from analysis of caustic soda and sodium chloride in the separated neat soap, analysis of sodium chloride and caustic soda in nigre and identification of turbidity in the nigre sight glass.

For in–line fitting and batch separation some analytical guidance is possible but in general the operator relies on traditional methods such as appearance to gauge the condition of soap. Using in – line fitting it is possible to fill soap pans brim full which represents a 20 – 30% increase in capacity of the pans in comparison with batch fitting. (For each fitting, space is left in the pans for boiling of the contents with open steam). After settling the content of the pan for 24 – 48 hours dependant upon required quality considerations, neat soap is removed by “skimming” the pan and the residual nigre and soap crust treated in the traditional manner by boiling, graining with solid salt and removing “weak” lyes. Weak lyes are incorporated in wash liquor while the residual soap in the pan can be treated in several ways to ensure that it is blended into the subsequent batch of soap.

SOAP RECEIVING PAN/TANK

It is advantageous to have a large pan/tank receiving the saponified soap which is continuously recycling as well as feeding forward the neat soap made. It is especially advantageous for SWING process plants as a means of

- Evening out the concentration variations over time
- Taking the saponification to nearly 100% complete by providing extra residence time

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