

A treatise of filter cake washing mechanisms in pressure and vacuum filtration systems

BARRY A. PERLMUTTER

BHS-Filtration Inc.
9123-115 Monroe Road
Charlotte, North Carolina 28270
Tel +1 704.845.1190
Fax +1 704.845.1902
barry.perlmutter@bhs-filtration.com

INTRODUCTION

During many chemical and pharmaceutical process operations, cake washing is required since no filter system can remove all of the mother liquor by pressure or vacuum dewatering only. The need for cake washing to produce a "clean cake" for further processing, drying or disposal can be for several reasons including the removal of the mother liquor, exchange of solvents, dissolution of soluble components and the displacement of insoluble contaminants.

The topic of "how to most efficiently wash the cake" is one that many process engineers struggle with when considering the various techniques available to meet the economic, production and quality requirements of the operation. Chemical engineering textbooks are notoriously shy on the subject and such information that is available is frequently lost in a host of theoretical equations. This article introduces pragmatism into cake washing theory and will help to guide engineers during their decision-making processes.

SELECTION OF EQUIPMENT

Before discussing cake washing, it is necessary to examine some of the equipment that can produce a cake, from thin-cakes (3-25 mm) to thick cakes (up to 300 mm). It can be suggested that any filter can produce a cake to be washed. Given an infinite amount of time and patience, yes perhaps this is true; the real question, however, is "does the process have enough time and do the operators have enough patience?"

The obvious choices for cake-producing filters that are also capable of washing the cake are well documented in the literature. These include rotary pan filters, "box" filters, filter presses, belt

filters, centrifuges, rotary drum filters and nutsche filters. Whether the application is, in principle, better suited for one rather than another depends upon many factors including the process requirements, cost, space availability, etc. The equipment selection should occur based upon the overall balance of the objectives and should include bench top and pilot scale testing.

However, to evaluate if the selected filter will meet the cake washing requirements, there is no substitute for painstaking laboratory tests simulating, in the best possible manner, the conditions of the pre-selected filter and then, based upon the washing results, decide if this filter remains the appropriate choice. If not, then the filter selection must be re-evaluated.

CAKE PREPARATION FOR OPTIMUM WASHING

It is a logical assumption, but in most cases erroneous, that the drier the cake the easier the cake can be washed. The reason for this apparent contradiction is either one or both of the following:

- If the cake is dewatered to its maximum through compression, the cake almost certainly will be so compact that no wash liquid can penetrate it. The wash liquid will look for the path of least resistance which means it will simply by-pass the cake or find the slightest crack and disappear through that crack.
- If the cake is dewatered beyond the dry-top by vacuum or blowing pressure gas (or steam) through it, the result will be the inevitable pockets (or cracks), which if anything will, once again, provoke bypassing.

In general, cake washing should begin immediately after the mother liquor has

published by **B5** srl
Via Cesare da Sesto, 10
20123 Milano - Italy
Tel. 0039 02 83241119
Fax 0039 02 8376457
www.b5srl.com

been drained away, leaving a well-settled, well-formed filter cake, which is saturated with mother liquor. If the surface of the cake (dry-top) shows signs of drying out, then it is usually too late.

WASHING MECHANISMS: DISPLACEMENT AND DILUTION

In almost all cases, the requirement for maximum wash efficiency is to develop a mechanism, which removes the required amount of mother liquor with a minimum of washing liquor. Basically there are only two mechanisms, and in practice usually both are present:

- Displacement of the mother liquor by washing liquor;
- Dilution of the mother liquor through mixing with washing liquors.

Displacement is by far the most efficient mechanism and under ideal conditions would cause the washing liquor to act as a piston driving the mother liquor out of the cake without any dilution. This is clearly utopia although a high degree of “piston or plug-flow effect” can be achieved with a careful preparation of the cake and a careful application of the wash liquor. The trick lies above all in the careful application of the washing liquor to the saturated face of the filter cake.

In open filters like belt filters or rotary drum filters, one can visually observe how far the wash application disturbs the cake surface and thus prevents good displacement. In closed filters this is more difficult and a degree of guesswork – and checking the result of these guesses – is inevitable.

If, as is very common, the washing liquor has a lower viscosity than the mother liquor one must be careful not to apply too much pressure (positive or negative) because the (usually) lower viscosity washing liquid will find any slight crack or “easy” passage between particles in preference to pushing the mother liquor.

Finally, cake washing will practically always alter the structure of the cake – change of polarity, change of viscosity, change in temperature, physical disturbance with the introduction of the washing liquor, etc. As a result there comes a point where displacement washing all but stops and a dilution process begins.

Dilution can occur in three different scenarios:

- If the liquors are **readily miscible** and the cake liquor – by now a mixture of mother liquor and wash liquor – is not too much occluded inside the particles, its crevices or its clusters, it should be a fairly

straightforward dilution process with a fairly predictable curve.

- If the liquors are reasonably miscible and the mother liquor has **no great affinity** to the solid particles, but is held inside the particle crevices or particle clusters, then a degree of agitation can help to “tumble” the cake a little and scrub the residual mother liquor off the particles. Obviously this agitation should only start after the “free” cake liquor has been washed away and when one is left with only the occluded liquor held in the pores of the particles or between particles. This agitation action is, of course, very difficult to imagine, for example, in filter presses or centrifuges. Two exceptions to this would be a multi stage pusher centrifuges that “tumble” the cake as matter of fact or a pressure plate filter with vibrating plates rather than spinning plates.
- If the mother liquor has **a great affinity** to the solids and is held inside the pores of the solid particles, usually the easiest and most effective washing system is to remove the cake from the filter, re-slurry it with clean washing liquid and allow it time to effect the desired mixing / diffusion and then to re-filter the slurry.

AN ALTERNATIVE FINAL WASHING SCHEME: CAKE COMPRESSION VERSUS DILUTION

Since dilution is proportional to the volume ratios, the efficiency in terms of residual mother liquor being removed drops dramatically as the cake becomes cleaner (less mother liquor). Assuming a wash volume equal to the cake liquor volume, one can expect at best a 50% reduction. Initially this may be quite high, but by the time the cake only holds 0.1% of mother liquor, a subsequent wash would only remove 0.05%. With the present trend to produce purer end products, there may come a point where extremely large amounts of wash quantities and time would be required to dilute the last traces to meet such purity levels. In these cases, there is great merit in investigating compression of this (almost pure) cake, with or without gas blowing, and mechanically forcing part of the remaining diluted liquor out of the cake.

APPLYING WASHING LIQUORS AND PITFALLS TO AVOID

The objective for washing is to subject

the entire cake face to an even and evenly pressurized liquid wash. Certain filters offer better flexibility than others to achieve this effect. Above all one has to avoid “channelling” both through the cake and across its surface. (Dye tests will show this clearly – see “laboratory tests” section). If channelling does occur, it may be necessary to investigate a different type of filter.

It must be clear, but is often ignored, that washing at too high a pressure differential only leads to poor efficiency. Too high a pressure almost always leads to bypassing. Although the ideal cake has an even structure, no pinholes, and no cracks and lies firmly bedded against the retaining walls, we are not living in an ideal world and to some degree all cakes will have these faults. Forcing liquid through the cake at unnecessarily high pressures only makes matters worse. A pinhole will become a hole, a crack a ravine and if the cake does not bed down perfectly, the washing liquid will make a highway of the gap. In addition, high pressure differentials reduce the contact time. In all cases therefore the optimum pressure differential is the lowest possible.

With (continuous-indexing) horizontal vacuum filters, one can use overflow weirs to gently pour the liquid onto the cake to avoid cake disruption.

On all filters, after an initial displacement wash, the subsequent diffusion / dilution washes may benefit from misting sprays which often give a better and/or even “wetting” effect. The problem here is, however, that fine sprays may have a tendency to block the cake structure, whereas the coarser sprays may tend to disturb the cake.

In this respect, engineers may custom-design a highly effective arrangement when trying to optimize a washing technique. One such customer designed a unique spray system for a continuous-indexing vacuum belt filter. The system used a square pattern spray, angled at approximately 45° and placed it so that the jets lost their velocity at just the point where washing should take place and where thus a gentle rain of washing liquid dropped onto the cake, as shown in Figure 1. This is by far both a very inexpensive and efficient / gentle wash liquid applicator system and can be used equally on rotary drum filters, belt filters, and rotary pan filters etc.

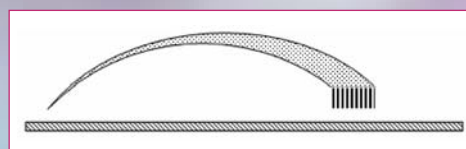


Figure 1 – Custom-design and highly effective washing arrangement

CO-CURRENT WASHING VERSUS COUNTER-CURRENT WASHING

As with filter selection, the decision for co-current or counter-current washing sometimes is based upon the history of the process. However, by careful analysis, the decision of co- or counter-current washing can be made based upon strict objectives.

The co-current system is clearly simpler to install and to operate. It is also possible that with some special attention to details an acceptable overall efficiency can be obtained. In addition the co-current washing can be carried out on most filters whereas with counter-current washing one has to use filters of a large linear configuration, which in most cases means horizontal vacuum belt filters. Continuous-indexing designs offer the maximum separation of filtrates and hence the maximum efficiency for counter-current washing.

A well-designed counter-current washing scheme for a filter can achieve remarkable savings in wash liquor usage along with the corresponding economical savings. Since the reason for contemplating counter-current washing is practically always an economical one, a very careful balance has to be made for the total capital investment and running cost of both a co-current and counter-current system. Almost inevitably the counter-current system will be more costly to install, will take up more space and will have a higher energy cost; whether or not this is justified by the process gains, can only be decided by the process application.

INSTALLING A COUNTER-CURRENT CAKE WASHING LOOP

The purpose of counter-current cake washing is to reduce as much as possible the quantity of washing liquid used for a given result. The reasons are practically always economical and for this reason it is even more important that the laboratory washing tests are being conducted under controlled and verified conditions. In most cases counter current washing is used on continuous equipment although it is perfectly feasible to do so on a number of batch filters, the principles remaining the same. A typical counter current washing train is shown in Figure 2, in this case using a continuous-indexing horizontal vacuum belt filter, which is probably the most commonly used filter type for good and controllable cake washing.

With the cake travelling from the left to the right, the clean washing liquid is introduced at the extreme right hand

side, is collected and via pumps re-introduced as a washing liquid now containing some of the mother liquor onto the stage immediately before the last one etc. The increasing line thickness indicates the increase in concentration in the washing liquors, whereas the crosshatched "cake liquor" is shown tapering towards the end of the filter, indicating the removal of mother liquor.

At the Feed End of the filter, the two liquors, the concentrated washing liquid and the mother liquor, are being fed forwards either combined or as individual streams. This is the classical scheme which is shown in just about every textbook, but which ignores a very important alternative. If, as is common, a degree of displacement washing

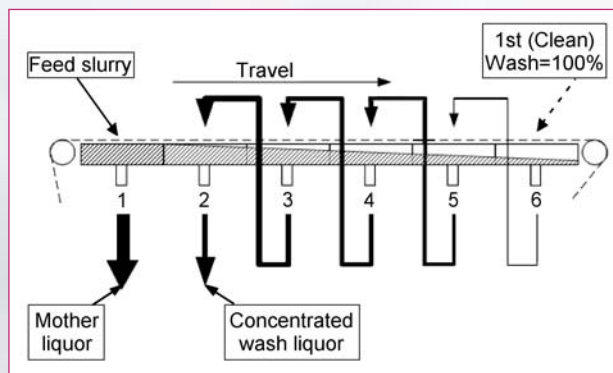


Figure 2 – A typical counter current washing train

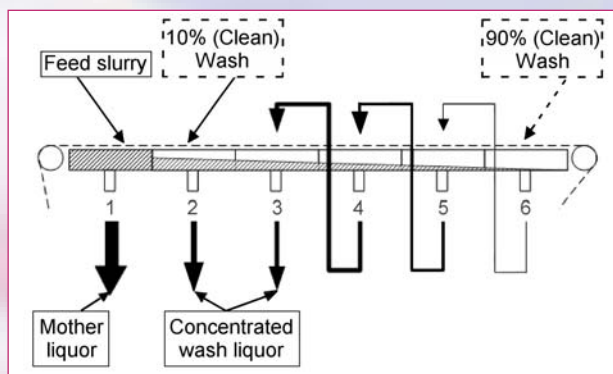


Figure 3 – 90% of the available volume of washing liquor is fed to the tail end of the filter as in Figure 2. However, the remaining 10% is introduced as a displacement wash immediately after dewatering

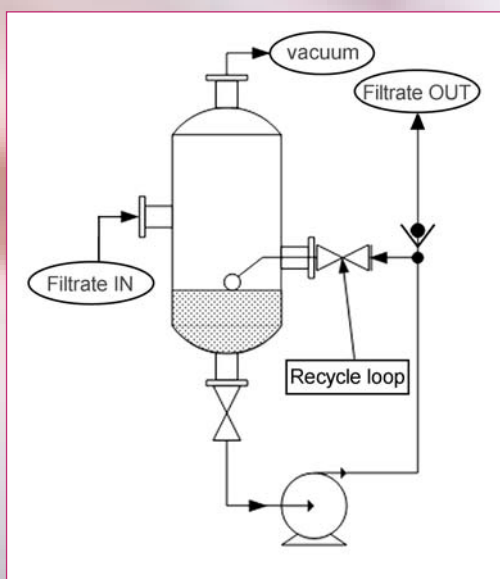


Figure 4 – Typical wash liquid recycle loop

can take place after initial dewatering of the cake, it would not be terribly logical to do so with a washing liquid, which already contains a high concentration of mother liquor. The resultant cake simply would not be very clean.

In Figure 3, there is shown the alternative approach, which in most cases gives much better efficiencies. In this case (an arbitrary) 90% of the available volume of washing liquor is fed to the tail end of the filter as in the previous case. However, the remaining 10% is introduced as a displacement wash

immediately after dewatering. This obviously leaves a much "purer" cake for the counter current washing stream, and thus can lead to either fewer steps or less washing fluid volume or both. It also eliminates one recycle loop complete with pumps, flow control etc.

In this respect it is important to remember that for the filter size the only thing that matters is the TOTAL hydraulic load on the filter and not the individual volumes, which are being recycled. If not watched carefully this can lead to excessively large filters.

For example, if in a co-current washing scheme, a cake can be washed with washing liquid equal to five times the volume of the Mother liquor in the cake, then the same cake can be washed with only 1.5 volumes in counter current fashion; but while the counter current loop consists of five stages, the total hydraulic load has increased by only 50% – $1.5 \times 5 = 7.5$ times, as opposed to 5 times. In addition each loop consists, external to the filter itself, of a receiver, a pump, level control, flow control and the pipe work to go with it. All of this can be a sizeable percentage of the total capital investment for the project as well as of the operating costs.

One important point is that it is essential that the counter current stream be introduced to the filter cake evenly

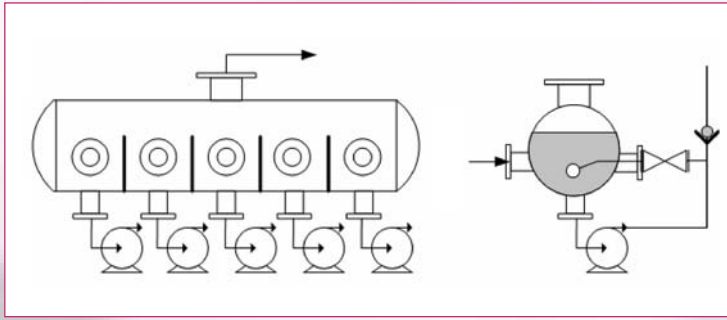


Figure 5 – Typical multi-compartmented receiver as an alternative to individual receivers shown in Figure 4

and in proportion to the volume introduced in the previous stage. This can lead to the installation of quite complex and expensive flow control.

To simplify this problem and reduce it to basic mechanics, a recycle loop, as shown in Figure 4, can be installed. It consists of a filtrate receiver, which in the case of vacuum filters is connected to a vacuum system or can be vented in case the filtrate drains from a pressure filter. A filtrate pump with an approximate 10% overcapacity runs continuously, delivering the filtrate to a Tee which connects with a float valve in the receiver and to a non-return valve and piping leading to the next washing stage. As soon as the pump overtakes the incoming filtrate, the float valve opens and the filtrate recycles preferentially back to the receiver, until the float valve closes and the filtrate goes to the next washing stage. In practice the float valve finds its own level resulting in an almost 100% constant flow of filtrate to the next stage. It is recommended that the riser above the non-return valve is 1 meter or more. If pipe work has been oversized then it may require a reducing valve above the non-return valve. This system has been used for more than 25 years in numerous installations and has always performed well, in spite of a complete absence of sophisticated control equipment.

Due to the fact that counter-current loops require a relatively large amount of space if individual recycling receivers are used, there is merit in using a single, multi-compartmented receiver as shown in Figure 5. Provided that all stages can operate at the same pressure differential, this arrangement is a design that can be used to minimize space and cost.

LABORATORY TESTS: TEST APPARATUS

How well a cake can be washed, if at all, depends 100% on the cake structure. Numerous articles have been written about cake construction or cake build-up. The theory is generally correct but in most cases only of academic interest, as the most important question that

interests process engineers is "our" cake and "our" process. What the process engineer must know is: "how well does this cake release

whatever type one has to use a filter chamber into which the suspension is pumped at the required pressure. All these types will produce cakes of different structures and thus have different behaviours.

DEWATERING / CAKE FORMATION

In the same way that an actual filtration curve is impossible to calculate without

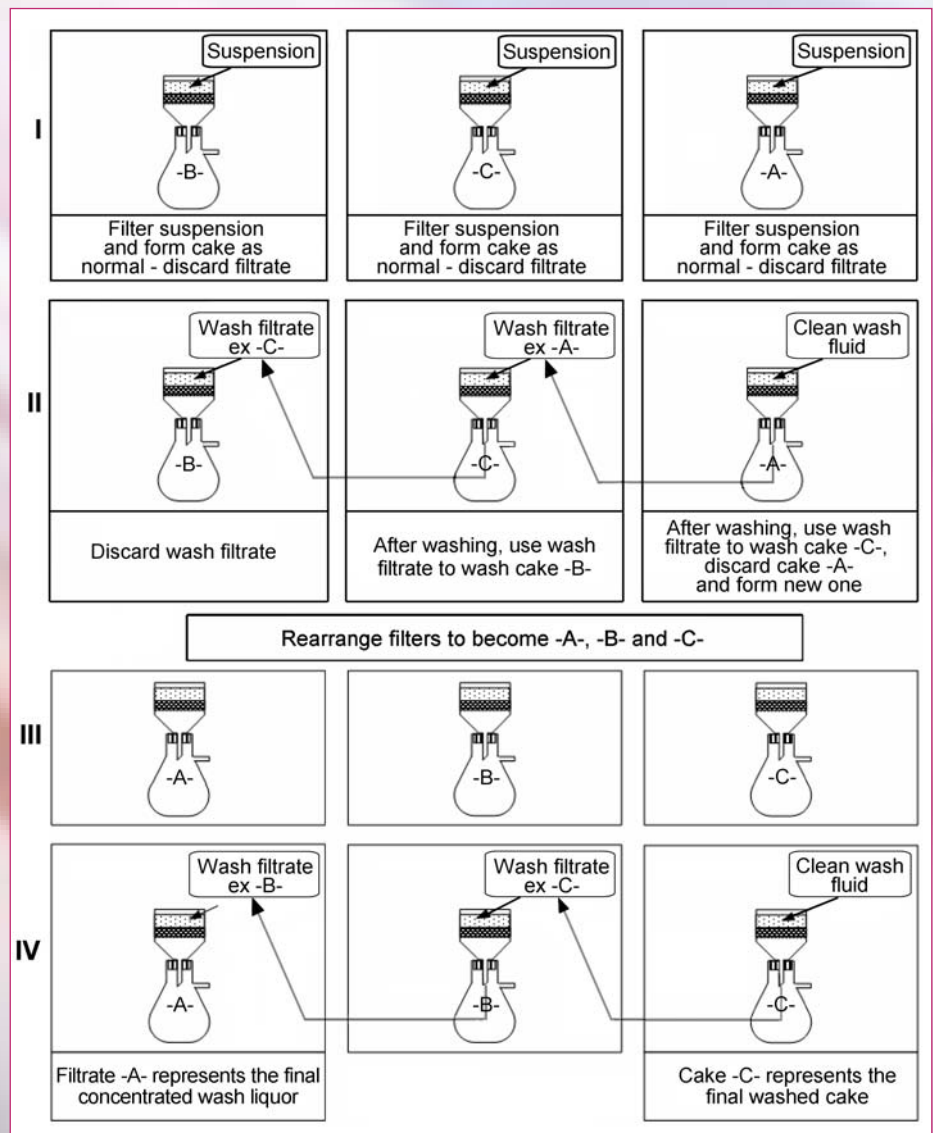


Figure 6 – The principle of the counter-current washing laboratory tests

its mother liquor under the influence of washing liquors?". Of course, much more important than theories about the cake, is the use of the correct test apparatus, which must be able to simulate the actual working of the pre-selected filter.

For example, if one thinks of a rotary drum filter, one has to use a filter leaf, which can be dipped upside down in the suspension while for a horizontal vacuum filter one can use a standard filter funnel. Continuing these thoughts, for a centrifuge one has to use a laboratory centrifugal filter and for a filter press of

having carried out basic filtration tests on the appropriate test equipment, cake washing is equally impossible to calculate without laboratory data. Any filtration and or washing tests that are conducted must produce filter cakes of the thickness, which one expects under actual plant conditions. If one expects to use a filter press producing 30 mm cakes but carries out washing tests on a 15 mm thick cake, one cannot be sure of the results.

As for the test apparatus, the size of the lab filter units can range from 50 mm (2 inches) up to 150 mm (6 inches) or

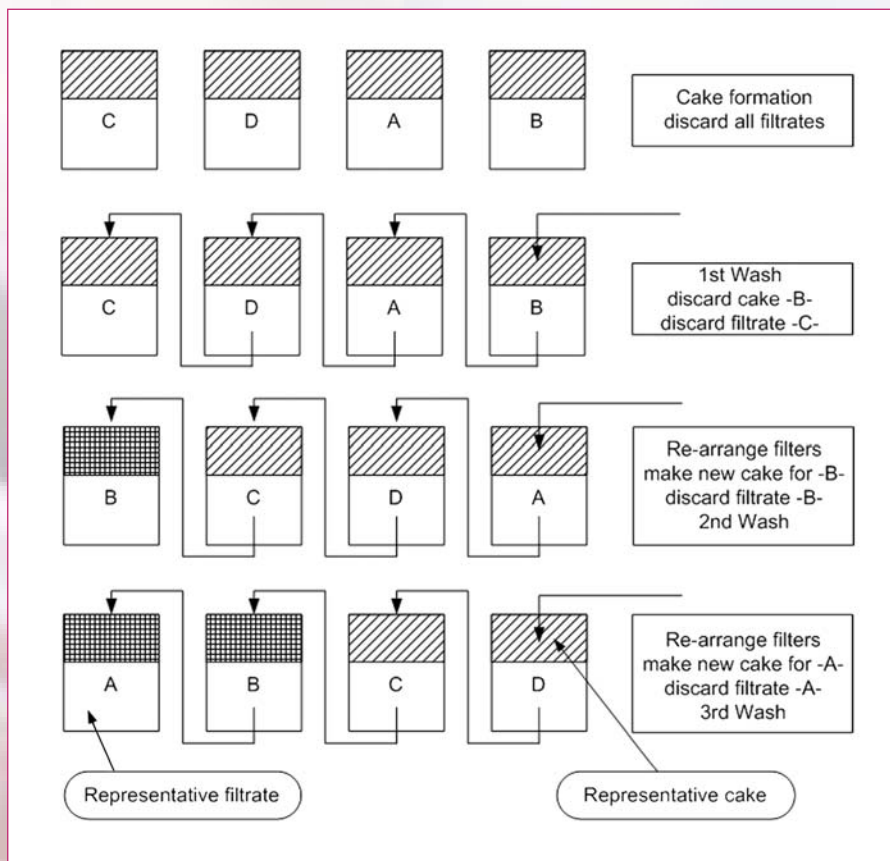


Figure 7 – A schematic for 4 stages of washing which can, of course, be further extended to 5-6 or more stages

even larger if space and suspension volumes permit. The edge effects, in small lab filters, can become a disproportionate factor giving highly misleading results. In addition if the cake has a tendency to crack, usually the result of shrinkage, this may not show up in a small unit, since there may not be sufficient mass to produce the shrinkage crack. It is critical that the engineer conducting the bench-top tests be an experienced filtration engineer from the specific vendor or from the operating company. Filtration experience, or the art of filtration, will have an influence upon the testing results.

For equipment with a given and reproducible cake thickness, the first stage of cake formation is fairly simple as there are not many possible variations; although the basic rule of all pressure filtration applies: Do not start immediately with a high pressure differential but build the pressure up slowly, allowing the cake to form evenly instead of being forced.

For equipment, which allows for different varying cake thicknesses, tests should be carried out at different thicknesses. The problem is that often the optimum cake for basic dewatering is not ideal for washing and vice versa. The reason for this is that a cake that drains easily and rapidly probably also passes the wash liquors rapidly without doing much mother liquor displacement.

Conversely, an ideal "cake wash thickness" may cause the overall filtration and washing time to be too long for practical purposes; some compromises, in the art of filtration, are often inevitable.

CO-CURRENT WASHING TESTS

After having established the basic filtration (dewatering) parameters, one can begin the painstaking process of introducing a given amount of wash liquor, draining the cake as far as possible, opening the filter and measuring the residual mother liquor in the cake.

One important factor is the procedure for introducing the wash liquor. If one uses a normal filter funnel (to simulate horizontal filters), then care must be taken to ensure that the washing liquor does not disturb the cake and its surface. At times this can be quite difficult and may require pouring the wash liquor along a spatula. An atomizing spray clearly helps greatly but it is more difficult to check the exact volumes which one sprays. An open (about 100 micron) monofilament cloth laid on top of the cake may protect the surface from being disturbed.

Measuring the mother liquor content in the filtrate is interesting but, in reality, is not a measure of cake washing efficiency. For example, a filtrate with a

low level of mother liquor can indicate that the cake is washed or that most wash liquor by-passed the cake and did not do any washing at all.

In terms of the test pressures, the tests should be repeated for, at a minimum of at least three different pressure differentials. Almost certainly one pressure differential gives better washing than another. If this indicates a trend, follow that trend to the optimum condition. Thereafter different volumes need to be tested and ultimately one can build up a washing profile, which then can be the basis for the actual plant conditions.

Because mother liquor determination can be time consuming, and above all, to get a better understanding of the wash pattern, it is a great help to introduce a soluble dye in the suspension – fountain pen ink being often the easiest. This gives an immediate visual indication of the displacement wash effect – the wash filtrate is dark blue, although the wash liquor is colorless – and the effectiveness of the subsequent dilution steps. When taking the cake out of the filter – without disturbing the cake if at all possible – one can also see clearly how effective the washing has been. If the cake shows areas of dark blue then clearly there has been either a distribution problem with the wash liquor or in these areas the cake was too compressed, preventing penetration with wash liquor. Equally white "veins" indicate that there were, perhaps invisible, cracks through which the wash liquor travelled and washed that part and not much else.

COUNTER-CURRENT WASHING TESTS

Before starting on a counter-current washing test, some co-current tests should be carried out to get a "feel" for the cake's behavior and the way the wash liquor removes the mother liquor, as well as for an approximate guess at the volumes required to obtain the desired results.

The principle of the counter-current washing laboratory tests is shown in Figure 6. As shown, three filter funnels are set up in one line. All funnels must have identical dimensions, have the same drainage grids, same filter cloth and be connected to the same vacuum source. They are marked A, B and C. To prevent confusion it helps to mark both the funnel and the flask. Sufficient suspension for four cakes must be available and be kept gently agitated and/or heated in case of hot suspensions. The initial order is B, C and

A. Following the instructions one finally obtains representative washed cakes and filtrates. Representative, that is, for a three-stage counter-current washing scheme.

It will be noted that this test does not allow for a 1st displacement wash with clean wash fluid. It is rather complex to introduce this 1st step in a laboratory set-up and since the efficiency of a first displacement wash can be measured exactly, it is relatively easy to allow for this in the final calculations, once the efficiency of the whole counter current train has been established.

Clearly one can expand the test to include as many stages as one likes although in general a three stage counter-current wash is fairly optimal. A schematic for four stages is shown in Figure 7 and can, of course, be further extended to 5-6 or more stages. During all of these tests all filtrate volumes, wash volumes, wash times and cake purity must be recorded. Finally, it may not be obvious from these schematics, but these tests are always more time

consuming than they first appear and therefore it is generally recommend that two trained operating engineers or the vendor and the process engineer perform the test work together.

CONCLUSION

Filtration and cake washing are affected by many variables. For example, in terms of the particles, the amount of solids, size, shape, particle density, compressibility of the solids, zeta potential and other ionic forces, agglomeration of the particles due to internal bonding and forces, etc. all impact the filtration flux rate and washing rate. Two particles of the same size, may behave differently if one is flat in shape while the other is irregularly shaped. In terms of the liquid, there are also characteristics that impact the filtration and washing rates such as temperature, viscosity, density, pH, components of the liquid, chemical additions (flocculants, coagulants, etc.), polar/non-polar constituents, the

interaction of the solids and liquids, etc. Once again, a small change in the liquid, can have a dramatic impact on the rates.

As can be seen, many variables must be examined during filtration and washing studies. This article discussed techniques to conduct washing laboratory and pilot studies and then how these results can be transferred to production systems. The process engineer through the development of an optimum test plan can analyze the resultant cakes and slurries to maximize his or her process. If the actual results differ from the tested results, then, with a good baseline of tests, the engineer can analyze the differences and develop the necessary corrective actions for a successful result and process.

ACKNOWLEDGEMENT

The author, President and Managing Director of BHS-Filtration Inc., would like to thank Mr. WIM PIERSON, minority owner and Director of BHS-Filtration Inc., for his help and assistance with this article.