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232 001

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Hydraulic Cyclone Study for Liquid-Solid Separation

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1.0 SUMMARY

Results are reported for studies of application of liquid cyclones or hydroclones to removal of 1 to 10 micron size particles from aqueous streams. Optimum dimensions were determined, and 0.25 in. and 0.40 in. diameter units were found to have capacities of 0.2 and 0.5 gpm for 80 ft of liquid pressure drops. For these dimensions and conditions, hydroclones would remove 50 percent or more of precipitated rare earth sulfates, ZrO_2 , or iron oxides (sp. gr. of ~ 2.5 to 5.) of 2 micron size from 250°C - 300°C aqueous streams. The studies with 0.16- to 0.50-inch-diameter hydroclones were used to extrapolate and test literature correlations of flow capacities and particle diameter efficiencies for hydroclones. Application of a method of batch underflow accumulation was developed and was found to give heavy phase concentration factors of up to 4,500. Titanium hydroclones were found to have corrosion rates of 10 mil/yr or less for 10 to 300 g U/Kg H_2O UO_2SO_4 solutions at 250 to 300°C.

2.0 INTRODUCTION

Studies were made to permit application of hydraulic cyclones or hydroclones to removal of 1 micron size solid particles from aqueous solutions. A simple, durable, liquid-solid separation apparatus was needed to remove precipitated fission and corrosion products from UO_2SO_4 solutions used in aqueous homogeneous reactors. The necessity of removing 1 micron particles requires hydroclones of less than one inch diameter. Experimental studies were limited to 0.16- to 0.50-inch-diameter units. The results were intended to permit selection of both optimum hydroclone units and optimum operating procedures for the proposed reactor applications.

The use of liquid-solid separators was suggested by the low solubility of some fission products in UO_2SO_4 solutions (4). Solubilities are in the range of 3 to 50 ppm at 250 to 300°C for many contaminants of reactor fluid streams. These include iron oxides and ZrO_2 in water and iron oxides, ZrO_2 , rare earth sulfates, and some other fission products in UO_2SO_4 solutions. Complete removal of the insoluble and the gaseous fission products would represent removal of $>80\%$ of the fission product poisons. Thus, a combination of liquid-solid separation and gas removal methods could reduce, and perhaps completely replace, other methods of processing aqueous solution fuels. Removal of insoluble corrosion products from aqueous coolants is another possible application of hydroclones to reactor systems.

In a hydroclone, the centrifugal field caused by the high velocity of fluid entering on a tangent results in accumulation of the heavy phase at the walls. Then it can be withdrawn as an underflow stream. A hydroclone has its feed port at the point of largest diameter (see Figure 1); this diameter is used to identify the hydroclone size. The vortex finder, extending down into the hydroclone cavity, contains the overflow port. The hydroclone body tapers to the underflow port. Experimental work reported by several investigators (2, 3, 5)

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Figure 1 Typical Hydroclone.

has determined the hydroclone flow patterns (see Figure 2). The fluid entering takes spiral paths downward. As the fluid moves toward the hydroclone center, at some point the vertical velocity reverses to a spiral path to the overflow port. A particle which is to be removed in the underflow stream must be accelerated by the centrifugal force to a velocity which keeps it out of the inner spiral to the overflow. Boundary flows apparently exist with such a flow at the top resulting in a short circuit of fluid with little phase separation.

The major advantages of the liquid cyclone or hydroclone are simplicity, small size, high efficiency, and high capacity. They can be easily designed to handle high-temperature and high pressure fluid streams. Almost any material which will withstand the fluid processed can be used as the material of construction. The energy required for the separation process is obtained as a fluid pressure drop and can be supplied by a pump. As compared to a centrifuge or continuous filter, a hydroclone has no moving parts and no mechanical seals. The concentrated solids leave as a slurry stream. A hydroclone avoids nearly all the difficult remote maintenance or cleaning problems associated with centrifuges or filters in radioactive applications.

3.0 HYDROCLONE DIMENSIONS

Studies were made to permit dimensioning of hydroclones to separate particles of 1 to 10 micron sizes, since precipitated rare earth sulfates and corrosion products were expected to be in this size range. Experimental work was limited to 0.16- to 0.50-inch-diameter hydroclones. Hydroclone efficiency comparisons were made by using a calcined ThO_2 slurry in H_2O . The ThO_2 was sized by several methods; one test material was 80% by weight between 0.6- and 3.0-micron-diameter. Efficiencies for this material ranged from 20 to 70 percent (see Table 1). The first four values show a comparison of the effects of the feed to overflow pressure drop. The remaining columns are for half a dozen different hydroclone units and show the reproducibility of results.

DIAMETER EFFICIENCY CORRELATIONS -- Constants were calculated for efficiency correlations developed from Stokes Law and from experimental studies reported in the literature (1, 5). The particle diameter removed with 50% efficiency, d_{50} , can be shown to be proportioned to $(D_c)^{1/2}$ and to $(\Delta P)^{-1/4}$. The following equation is suggested for rough estimates of the performance of hydroclones of less than one inch diameter and with the optimum dimension ratios determined (see Table 2).

$$d_{50} = 5 \times 10^3 \frac{D_c^{0.5} \mu^{0.5}}{\Delta P^{0.25} (\rho_s - \rho_l)^{0.5}} \quad (1)$$

d_{50} is the particle diameter in microns removed with 50 percent efficiency

D_c is the hydroclone inside diameter in inches

μ is liquid viscosity in lb/ft sec

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Fig. 2. Hydroclone Flow Patterns.

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Table 1. Hydroclone Efficiency Studies

Material: Thorium oxide in water at room temperature. By wt. percent, 80% of ThO_2 between 0.6 and 3.0 micron diameter.

<u>Hydroclone Dimensions</u>		<u>Feed Conditions</u>		<u>Underflow Ratio</u>		<u>Hydroclone Efficiency</u>
<u>Diameter</u> <u>D_c (inches)</u>	<u>Length</u> <u>L (inches)</u>	<u>$P_F - P_O$</u> <u>(psi)</u>	<u>Flow</u> <u>(gpm)</u>	<u>$\left(\frac{B}{F}\right)$</u> <u>(%)</u>		<u>E_1 (%)</u>
0.25	1.50	25	0.15	6		27
		49	0.20	6		36
		136	0.30	6		57
		251	0.40	6		67
0.25	1.50	35	0.22	6		42
		35	0.21	6		43
0.40	3.14	35	0.72	6		37, 34, 40
		35	0.77	6		43
		44	0.87	9		37
0.40	2.40	44	0.76	10		39, 44, 42

Table 2. Hydroclone Dimension Ratios

Basis: Studies on hydroclones of less than one inch diameter. Values are consistent with literature values for 3-6 in. diameter hydroclones

Dimension		Suggested Ratios As Fraction of D_c Dimension	Combination For Use With An Underflow Pot
<u>Symbol</u>	<u>Nomenclature</u>		
D_c	Hydroclone i.d. at feed port	1	1
L	Hydroclone inside length	5-8	6
$D_F^{(a.)}$	Feed port diameter	$1/3-1/7$	$1/4$
D_u	Underflow port diameter	$1/3-1/6$	$1/4$
D_o	Overflow port diameter or vortex finder i.d.	$1/4-1/6$	$1/5$
OD_o	Vortex finder o.d. - extension into hydroclone	$1/3-1/6$	$1/4$
L_o	Vortex finder length - extension into hydroclone	$1/3-1/2$	$3/8$

a. Use of a rectangular feed port is usually desirable. Then the port area may be made equivalent to circular ports of the diameters suggested.

ΔP is the feed to overflow pressure drop in feet of liquid

ρ_s is the solid phase density in lbs/ft³

ρ_l is the liquid phase density in lbs/ft³

The particle diameter, viscosity, and density terms are fixed by the system to be processed. The hydroclone diameter and the pressure drop can be varied to obtain the necessary hydroclone efficiencies. For small particles (~1 micron diameter or less), agglomeration can result in much higher efficiencies than would be expected from the true particle size.

DETERMINATION OF OPTIMUM DIMENSIONS --- Optimum hydroclone dimensions for 0.25 and 0.40 inch diameter units were determined using the ThO₂ slurries for comparison studies. These optimum dimensions can be expressed as ratios of a primary dimension; this was done using the inside diameter of the hydroclone at the feed port, D_c , as this primary dimension (see Table 2). These optimum dimension ratios were based on the efficiencies for pressure drops of 80 ft of liquid. Since these ratios agree with literature values for larger hydroclones, they probably apply to a wide range of sizes. The hydroclone length, L , feed port diameter, D_f , and overflow port diameter, D_o , showed distinct efficiency maximums. The length and the underflow port diameter determine an induced underflow ratio to be discussed (see Figure 4). Use of a cylindrical section at the point of feed introduction showed no detectable advantages and was omitted to simplify hydroclone construction.

FLOW CAPACITY CORRELATION -- For dimensionally similar hydroclones, the flow is proportional to the feed to overflow pressure difference to the one-half power and to the hydroclone diameter squared. A more generalized correlation has been proposed by Yoshioka and Hotta (5).

$$\Delta P = \frac{K Q^2}{D_c^{0.9} D_f^{1.2} D_o^{1.9}} \quad (2)$$

ΔP is the feed to overflow pressure difference

Q is the volume feed rate

D_c is the hydroclone inside diameter

D_f is the equivalent feed port diameter, i.e., the diameter of a circular port of equal area.

D_o is the overflow port inside diameter

K is a constant for a selected set of units

The value of K would actually be a function of the fluid system, the surface finish, and perhaps of other hydroclone dimensions. Some values of K were determined experimentally or from literature data for feed flow in gpm, diameters in inches, and pressure differences in feet of liquid (see Table 3). The values of K apply for 80% or more of the feed volume leaving through the overflow port. The causes of the variations in K are not well enough known to justify a re-correlation. When a hydroclone is designed with the dimension ratios previously suggested as being optimum (see Table 2), the diameter terms in (2) can be

combined into one variable. The equation for hydroclones of less than one inch diameter then becomes:

$$Q = 0.36 (\Delta P)^{0.5} D_c^2 \quad (3)$$

4.0 BATCH UNDERFLOW ACCUMULATION

Accumulation of the heavy phase in an underflow receiver was found to be a useful alternate method of hydroclone operation. The receiver or underflow pot is a cavity into which the hydroclone underflow port opens (see Figure 3). The concentrated heavy phase flows through the underflow port along the hydroclone walls. An equal volume of pot solution returns through the center region of the underflow port. This type of operation resembles the dust collection of a gas-solid cyclone separator. Settling out in the underflow pot could increase the effectiveness of a hydroclone using this batch type of operation. However, precipitated fission products collected by a hydroclone might refuse to settle out due to their small particle size and the presence of thermal convection currents from radiation heating.

This batch underflow accumulation eliminates an otherwise difficult flow control problem. With a continuous underflow, obtaining high concentration factors requires a small volume underflow rate. Metering this slurry stream would be a difficult problem. This metering would be especially difficult if the slurry was to be letdown from 100 to atmospheric pressure as would be the case in several proposed aqueous homogeneous reactors. The batch underflow accumulation permits isolation of the hydroclone and pot with batch letdown of the complete hydroclone and pot volume without disturbing the reactor operation.

The ratio of the volume flows of the induced underflow and the hydroclone feed is determined by the geometry and in turn helps determine the equilibrium concentration factor. Induced underflows of 0 to 4 percent by volume of the feed flow were possible without large changes in hydroclone efficiency. The values of hydroclone dimension ratios suggested (see Table 2) give induced underflow ratios of about 1-1/2 percent. A material balance can be arranged to give an expression for the equilibrium concentration factor. This concentration factor is the concentration returning to the hydroclone from the underflow pot over the feed stream concentration. At equilibrium, the feed and overflow concentrations will be equal.

$$CF = \left(\frac{F}{B}\right) \left(\frac{E_1}{1 - E_2}\right) \quad (4)$$

$\frac{B}{F}$ is the underflow to feed volume flow ratio

E_1 is the hydroclone efficiency for removal of heavy phase from the feed stream

E_2 is the hydroclone efficiency for returning to the pot any heavy phase in the volume displaced from the pot.

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Fig. 3. A Hydroclone and Underflow Pot.

Table 3. Values of K for Hydroclones

<u>Hydroclone Diameter (Inches)</u>	<u>K (Ft of Liquid)(in)⁴ (GPM)²</u>
0.16	0.060
0.25	0.062
0.40	0.07
3.0 to 6.0 ^(a)	0.15
3.0 ^(b)	0.15

(a) Reported by Yoshioka and Hotta.

(b) Calculated from values reported by Kelsall

Since E_2 approaches unity for materials for which E_1 is reasonably large ($> 30\%$), the possible concentration factor (C.F.) may be on the order of 10^3 or 10^4 . Concentration factors of up to 4,500 have been obtained experimentally (see Table 4). The efficiencies for precipitated iron oxides have consistently been higher than could be predicted from micro-photographs of particle sizes. This may be due to the particle agglomeration previously mentioned.

5.0 OPERATING PROBLEMS

CORROSION -- Acceptable materials of construction for hydroclones to be used at 250 to 320°C would be a 316 type stainless steel for H_2O and titanium for UO_2SO_4 solutions. Titanium hydroclones have shown corrosion rates of less than 10 mpy for UO_2SO_4 solutions of from 10 to 400 g U/kg H_2O at 250 to 300°C. (See Figures 4, 5.) We believe that titanium hydroclones will withstand any of the UO_2SO_4 concentrations presently proposed for aqueous homogeneous reactors. Stainless steel hydroclones tested with 10 to 15 g U/kg H_2O UO_2SO_4 solutions at 250 to 320°C have shown corrosion rates of less than 10 mpy up to over 100 mpy. The variations are probably due to minor variations in conditions since stainless steels have critical velocities which separate regions of high and low corrosion. The hydroclone port or wall velocities are on the order of 20 ft/sec. We consider stainless steel hydroclones undependable for UO_2SO_4 solutions although they are probably adequate for coolant water streams.

OPERATING FLEXIBILITY -- Commercial applications of hydroclones have included paralleling of units to obtain larger capacities and use of units in series to increase the concentration factors. These adaptations would also be possible for the proposed applications to reactor systems. Parallel units used for the batch accumulation type of operation might be able to use one common underflow pot. This type of operation will be tested to determine if corrosion or solids accumulation can result in zero underflow and thus zero efficiency for one of the parallel units.

No plugging of feed or overflow ports was observed during application of hydroclones to a variety of precipitated corrosion and fission products in high temperature, high pressure systems. The port sizes were on the order of 0.05 to 0.10 inch diameter. The possibility of plugging could be minimized by use of a coarse screen or scalping hydroclones to catch particles larger than the hydroclone port diameter. The underflow ports did not plug for induced underflow operation. When the underflow was temporarily interrupted for hydroclones designed for continuous underflow takeoff; the plugs of solids which accumulated in the underflow line or port were difficult to remove and tended to result in hydroclone erosion (see Figure 6).

A design was developed to permit replacement of a hydroclone in a radioactive system by use of a flanged closure and remote tools (see Figure 7). All connections are in the lower flange including the underflow pot on which the flange is mounted. The flange bolts, the hydroclone retainer, and the hydroclone could be removed and replaced by remote tools. This replacement might be required by a corroded or plugged hydroclone, or might be to obtain a different capacity, underflow ratio, or efficiency.

12

Fig. 4. Corrosion of a 0.40-in.-dia. Titanium-Lined Hydroclone.

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Table 4. Experimental Concentration Factors for
Underflow Pot Operation

Hydroclone Diameter (Inches)	Material	Material Size			Observed Concentration Factor $\frac{C_{pot}}{C_{feed}}$
		$50\% \leq d \leq 50\%$ (Microns)	Wt. %	in Size Range Range (Microns)	
0.25	ThO ₂	4.1	70	3.0 to 7.1	2300
		4.1	70	3.0 to 7.1	2420
		1.8	71	0.8 to 4.2	78
		1.8	71	0.8 to 4.2	93
0 0.40	ZrO ₂	1.0	80	0 to 4.0	4500
	Fe ₂ O ₃ ^(a)	0.5	80	0.2 to 0.5	270 ^b

(a) Observed CF and particle size measurements were for different samples of Fe₂O₃

(b) This CF is based on the total iron in the hydroclone feed and would be higher if it were based on insoluble iron only.

14

DIAMETER: 0.25 in.
LENGTH: 1.50 in.
MATERIAL: TITANIUM 55A

CONDITIONS: 10 g OF U/kg OF H₂O, 300°C
TEST PERIOD: 3975 hr
FEED FLOW: 0.23 gpm

Fig. 5. Corrosion of a 0.25-in.-dia. Titanium-Lined Hydroclone.

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15

DIAMETER: 0.40 in.
LENGTH: 3.14 in.
MATERIAL: 347 STAINLESS STEEL

CONDITIONS: 15 g OF U/kg OF H₂O, 250°C
TEST PERIOD: 200 hr
FEED FLOW: 0.6 gpm
REMARKS: $\frac{3}{4}$ -in. DEPTH PLUG OF SOLIDS
ABOVE UNDERFLOW PORT

Fig.6. Corrosion — Erosion of a 0.40-in.-dia. 347 Stainless Steel Hydroclone.

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Fig. 7. Cross Section of Integrally-Flanged Hydroclone.

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6.0 CONCLUSIONS

The conclusions from these hydroclone studies were as follows: The use of hydroclones is a practical method of liquid-solid separation which can be applied to high temperature-high pressure reactor coolant or fuel solution streams. As compared to presently developed centrifuges or filters, hydroclones would appear to be much more durable and trouble-free. The 0.25 and 0.40 inch diameter units tested effectively concentrated solid particles as small as one micron in size. The criteria for dimensioning hydroclones were extrapolated and tested down to 0.25 inch diameters. The high concentration factors desired for reactor applications can be obtained by batch accumulation in an underflow receiver. Titanium is an acceptable material for hydroclones to be used for processing aqueous UO_2SO_4 solutions at high temperatures while 347 stainless steel is not dependable.

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