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Design aspects of pulsed sieve plate columns

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Abstract

The design aspects of pulsed sieve plate columns have been studied. Various correlations presented in the past literature for flooding, drop size, hold-up and mass transfer coefficients have been analyzed. An attempt has been made to screen the available correlations, to select a correlation which can be applied with a large degree of confidence. The correlations were tested against a large set of experimental data gathered from the work of different investigators over the past 50 years. It consists of 400 data points for flooding, 104 for drop size, 247 for hold-up and 274 for mass transfer in pulsed sieve plate columns. The data covers a wide variation in geometrical and operating parameters of the pulsed sieve plate column and physical properties of the liquid–liquid systems. The exercise has helped in identifying correlations that can be used for design of pulsed columns. For mass transfer, none of the correlations reported in the literature were found to be satisfactory. The difficulties associated with this observation have been discussed. The results of the present study can be used to reduce the experimental work associated with the scale-up of the pulsed sieve plate columns.

Keywords: Pulsed sieve plate column; Flooding; Drop size; Hold-up; Mass transfer; Design

1. Introduction

Van Dijck [1] was one of the first persons to report a pulsed sieve plate column for liquid–liquid contacting. It has found wide applicability in nuclear fuel reprocessing. The irradiated fuel from the nuclear energy reactors contains a mixture of uranium and plutonium along with a large number of radioactive elements. It is beneficial from both economical and environmental aspects to recover the uranium and plutonium and reduce the radiation level in the stream before discharging it. Pulsed sieve plate columns have been used in this field for many years. Pulsing improves the rate of mass transfer by reducing the size of drops. Wiegandt and Von Berg [2] have shown that the height of a packed column required to effect a given degree of extraction is reduced by a factor of three, under pulsing conditions. The pulsed columns have a clear advantage over other mechanical contactors when processing corrosive or radioactive solutions since the pulsing unit can be remote from the column. The absence of moving mechanical parts in such columns obviates frequent repair and servicing. These advantages have lead to the application of these columns in chemical, biochemical and petroleum industries besides nuclear fuel reprocessing. Due to compact design, versatility and ease of operation these columns have been of interest to different investigators. This has resulted in generation of a large amount of experimental data on its hydrodynamics and mass transfer performance. Tables 1–3 summarize the operating and geometrical conditions for different investigators, physical properties of the systems used and parameters studied, respectively.

Most of the studies have resulted in empirical correlations. These correlations are important as they directly relate the performance of the column to its geometrical and operating parameters and physical properties of the liquid–liquid systems. However, the presence of a large number of correlations causes considerable ambiguities in choosing one correlation over the other. This leads to difficulty when one intends to design a column for a required extraction duty. Hence, a thorough analysis of these numerous correlations is required to screen those correlations which can be directly used for design. However, very little attempt has been made in the past to address this issue. In view of this, the following objectives were set forth for the present work:

(1) Analyze the information available on the hydrodynamics and mass transfer characteristics of pulsed sieve plate extraction columns.

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Nomenclature

$\underline{a}$	interfacial area (m^2/m^3)
A	amplitude of pulsation (m)
b	deceleration suffered by drop once it crosses the vena contracta (m/s^2)
c_o	orifice constant = 0.6
c_D	drag coefficient
d	hole diameter (m)
D	column diameter (m)
E_k	kinetic energy of drops (J)
E_σ	cohesion energy of drops (J)
f	frequency of pulsation (Hz)
H	column height (m)
h	plate spacing (m)
P	pitch of the holes (m)
V_1	velocity of drops in the region of sieve trays (m/s)
V_2	velocity of drops in the region away from sieve trays (m/s)
V_c	superficial velocity of continuous phase (m/s)
V_d	superficial velocity of dispersed phase (m/s)
V_0	characteristics velocity of drops (m/s)
V_∞	terminal velocity of single drops (m/s)

Subscript

a	mean value
c	continuous phase
d	dispersed phase
f	value of a parameter at flooding
g	acceleration due to gravity (=9.81 m/s^2)
m	maximum value of a parameter
p	possible value of a variable
t	transition between different regime

Greek letters

Δ	average velocity of all material past the pulse plates averaged over the entire pulse cycle (m/s)
α	fractional free area
ε	power dissipated per unit mass of fluid (W/kg)
ϕ	hold-up
λ	ratio of velocity difference to π times the pulse velocity
μ	viscosity ($N\ s/m^2$)
π	average velocity of all material past the pulse plates averaged over the actual time of flow in a direction (m/s)
π_M^2	root mean square of π_c and π_d (m/s)
π_v	average of π_c and π_d (m/s)
ρ	density (kg/m^3)
$\Delta\rho$	density difference between two phases (kg/m^3)
σ	interfacial tension between two phases (N/m)
ψ	$Af/(\beta h)^{1/3}(\mu_d^2/\sigma\Delta\rho)^{1/4}$ ($m^{11/12}\ s^{-1}$)

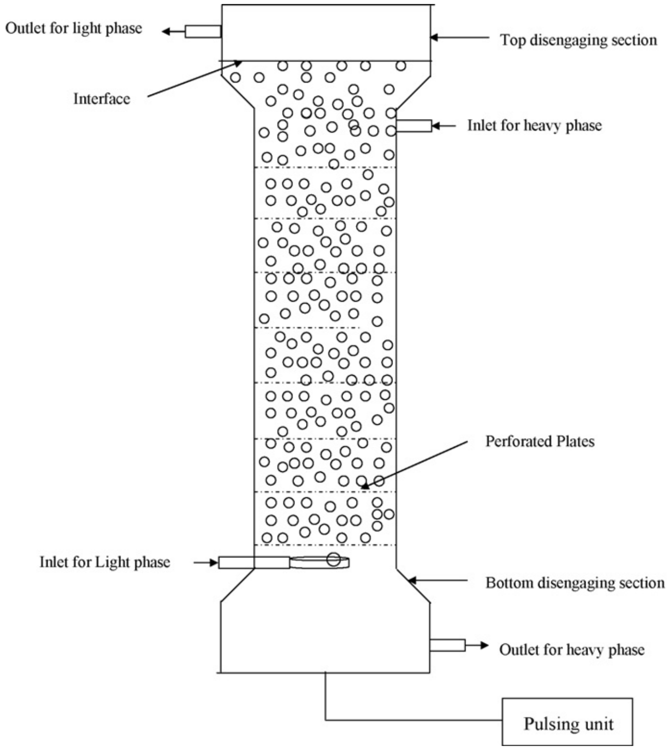

Fig. 1. Schematic of pulsed sieve plate column.

(2) Gather a large set of experimental data from the work of different investigators ensuring a wide variation in physical properties, geometrical and operating conditions of these columns.
(3) Test the applicability of various correlations reported in literature for flooding, drop size, hold-up, mass transfer and identify correlations which can be used by process design engineers for design and scale-up purposes.

2. Previous work

2.1. Operating principle and flow regimes in pulsed sieve plate columns

Pulsed sieve plate column consists of a cylindrical column fitted with perforated plates (Fig. 1). It uses mechanical energy in the form of pulsing. Pulsing displaces the layer of heavy liquid resting on each plate and the layer of light liquid collected under the plate. On the up stroke, the displaced volume of light liquid is forced through the holes in the form of jet streams into the heavy liquid above (Fig. 2). On the down stroke, the reverse process takes place, with the heavy liquid jetting downward through the light liquid. For constant phase flow rates there are three stable (mixer-settler, dispersion and emulsion) and one unstable regime of operation (Fig. 2) and two flooding types depending on the pulse frequency and amplitude as observed by different investigators [3–6]. Sege and Woodfield [3] defined flooding in pulsed sieve plate columns as the flow condition when the fluid of one phase entering at one end of the column can not leave at the opposite end and must exit through the out-

Table 1

Experimental studies on pulsed sieve plate columns: details of operating conditions and column dimensions

Investigators	Column dimension		Plate details				Pulse details			Phase velocities	
	$D \times 10^3$ (m)	H (m)	$d \times 10^3$ (m)	α	$h \times 10^3$ (m)	MOC	f (Hz)	$A \times 10^3$ (m)	$fA \times 10^3$ (m)	$V_c \times 10^3$ (m/s)	$V_d \times 10^3$ (m/s)
Cohen and Beyer [39]	25.4	0.51	1	9	50	Lucoflex	0.28, 0.59, 1.2	2.5–15.2	0.7–18.24	0.33, 0.82, 1.64	0.33–4.93
Sege and Woodfield [3]	76.2	2.74	1.5, 3.1, 4.7	10, 21, 23, 40	35.56, 50.8, 76.2. 101.6	Fluorothene, SS	0.5–1.3	12.7–38.1	16.93–33.86	1–28.6	1.8–35.1
Chantry et al. [49]	40	1.2	1.2–2	2–6	76.2	–	0.3–1.5	1.5–6.0	0.45–9	0.66–1.6	0.6–1.43
Li and Newton [50]	50.8	0.6	1.6	8.51	57.5	Brass	0–1.83	36	0–66	10.67–16.10	8.53–11.41
Thornton [13]	76.2	1.07	1.5, 3.0	13–60	12.5–50	Brass	2–7	2.8–18	5.6–126	0–10.6	0–4.67
Logsdail and Thornton [40]	152.4, 228.6, 304.8	1.8	3.0	25	50	SS	3	6.35–15.24	18–46	1.2–8.5	0.78–5.02
Sobotic and Himmelblau [51]	50.8	–	3	16.72	38.1	SS, Polyethylene	0.27–1.95	12.5–25.4	6–55	2.25–4.60	2.56–4.91
Smoot and Babb [52]	50.8	1.52	1.6, 3.2	23	40.3, 55.7	SS	0.5–1.5	12.8–25.4	6–150	2.2–5.76	2.2–6.0
Jones [22]	25.4	0.92	1.6, 3.175	0.328	50	SS	0.21–3.33	6.5–44.7	5.4–21.6	2.5–9.64	0.49–1.14
Sehmel and Babb [5]	50.8	2.15	3	23	50.8	SS	0.3–3	6.35–51.6	1.90–154	1.74–4.36	1.74–4.36
Miyauchi and Oya [9]	54	0.37–0.86	3	0.19	30–70	SS	3	1.7–15	5.1–45.0	2.7	0.43
Kagan et al. [15]	56	5.4	2	8.2	50	SS	0.8–1.67	5–15	4–25	6.2–6.7	0.6–1.2
Angelino et al. [24]	40	1.17	3	23.61	50	SS	0.17–2	5–25	8–50	4.44	0.9
Mishra and Dutt [41]	76.2	–	4.76, 6.35	0.226, 0.36	50, 100	SS	–	–	8.34–30.3	0.8–2.7	0.8–2.5
Bell and Babb [42]	50.8	1.2	3	23	56	SS	0.3–3	12.8–46.2	3.84–141	0.0–4.63	1.74–4.63
Khemongkorn et al. [29]	50	1	2	0.188	50	SS	1.5–3.0	20	30–60	5.66	0.14–0.28
Berger et al. [6]	80	5	4, 6, 8	23, 40, 60	50	–	0–4	8	0–50	2.35–6.7	1.35–9.7
Boyadzhiev and Spassov [10]	50	1	2	0.198	50	SS	2.–3	10–35	30–72	2.90	1.50
Berger and Walter [17]	80	5	3, 4, 6, 5	23, 40, 60	50	SS	0–3.33	8	0–24	1.6–5.5	1.1–7.8
Pietzsch and Blass [31]	72	3.5	4	0.26	50	SS	–	–	5–40	2–6.1	2–6.1
Kleczek et al. [32]	67	0.7	3	0.067–0.134	50	–	–	–	8–21	4.25	4.25
Lorenz et al. [18]	80	4.3	2, 4, 6	0.23, 0.39, 0.49	100	SS	–	–	0.7–21	6.1	6.1
Bahmanyar et al. [53]	76	0.43	3.18	22	50	SS	1	8–16	7.7–15.9	Single drop rising in stationary liquid	
Mohanty and Vogelpohl [37]	80	4.3	4	0.39	100	SS	–	–	15–20	47–61	47–61
Srinivasulu et al. [35]	43	2	3–8	0.28	50, 100	SS	–	–	22–78	3.15–7.2	1.05–5.02
Venkatnarasaiah and Verma [45]	43	2	3–8	0.28–0.46	50–200	SS	–	–	7–62	3.15–7.2	1.05–5.2
Luo et al. [36]	150	2	3	23	50	SS	1–2	6.7–20	6–35	0.6–2.4	1.3–4
Vatanatham et al. [55]	50	2.45	2	25	50	–	0.8–2.3	12–40	9–80	–	–
Li et al. [56]	150	2	–	23	50	SS, PTFE	–	–	10–32	1.347, 2.175, 2.761	1.5–4.5
He et al. [46]	600	6	–	–	–	–	0.92–1	2.85–6.5	2.85–6.0	0.245–0.451	1.472–2.355
Luo et al. [54]	150	2	3	0.23	50	SS	–	–	15–30	1.347	1.347–4.5
Tang et al. [64]	150	2	3	23	25	SS PTFE	–	–	10–32	1.347, 2.175, 2.761	1.5–4.5
Tribess and Brunello [19]	39.6	2.67	3	23	25, 50, 75, 100	SS	0.5–3.5	8	4–25	0.6–16	0.3–7.5
Usman et al. [63]	50	4.1	3	13	50	SS	0–2	20	0–40	1–8	1–8

Table 2
Experimental studies on pulsed sieve plate columns: details of liquid–liquid systems investigated and physical properties

Investigators	Continuous phase (c)	Dispersed phase (d)	Solute	Mass transfer direction	ρ_c (kg/m^3)	ρ_d (kg/m^3)	$\mu_c \times 10^3$ (Pa s)	$\mu_d \times 10^3$ (Pa s)	$\sigma \times 10^3$ (N/m)
Cohen and Beyer [39]	Water	*iso*-Amyl alcohol	Boric acid	d → c	997.1	822	0.8937	4.8812	4.58
Sege and Woodfield [3]	Aqueous HNO_3	12.5–30% TBP with CCl_4 or kerosene type liquid	$UO_2(NO_3)_2$	c → d, d → c	–	–	–	–	–
Chantry et al. [49]	Water	MIBK, ethyl acetate	Acetic acid	c → d	996.38	796.13, 900	0.998	0.554, 0.46	10.3, 6.8
Li and Newton [50]	Water	Toluene	Benzoic acid	d → c	998.2	866.7	1.003	0.586	35.4
Thornton [13]	Water	Toluene	Acetone	c → d, d → c	998	864	1.0	0.585	34
		Butyl acetate				881		0.748	14.2
		Ethyl acetate				905		0.490	8.5
		iso-Octane				805		0.614	10.1
		MIBK				691		0.486	40.0
		White spirit				772		0.926	36.0
Logsdail and Thornton [40]	Water	Toluene	Acetone	d → c	998	864	1.0	0.585	34
		White spirit				772		0.926	36.3
Sobotic and Himmelblau [51]	Water	MIBK	Acetic acid	c → d, d → c	998	691	0.987	0.486	40.0
Smoot and Babb [52]	Water	MIBK	Acetic acid	d → c	1000	800	0.998	0.525	9.0
		1,1,2-Trichloroethane	Acetone			1436		0.964	26.1
Jones [22]	Water	MIBK	MIBK	d → c	998	798.4	1	0.58	10.1
Sehmel and Babb [5]	Water	Hexane, benzene, MIBK	–	–	996.38	651.96, 871.42, 796.13	0.8420	0.2873, 0.58, 0.5544	38.745, 32.13, 10.325
Miyauchi and Oya [9]	Water	MIBK	–	–	996.38	796.13	0.842	0.554	10.3
Kagan et al. [15]	Water	Kerosene	–	–	1000	788.69	1	1.7584	39.0
Angelino et al. [24]	Water	Di-*iso*-propyl ether	Phenol	c → d	999	720	0.980	0.380	17.1
Mishra and Dutt [41]	Water	Toluene	–	–	998	863	1.0	0.586	33.5
Bell and Babb [42]	Water	MIBK	–	–	998.0	807.34	0.9779	0.4919	10.22
		Hexane				659.97		0.2939	46.55
Khemongkorn et al. [29]	Water	CCl_4	Iodine	c → d, d → c	1000	1590	1	0.95	45.0
Berger et al. [6]	Water	Toluene	Acetone	d → c	998	870	0.998	0.50	36.1
		Ethyl hexanol	Acetic acid			830		9.28	12.1
		n-Butanol				858		3.36	1.8
Boyadzhiev and Spassov [10]	Water	Kerosene + CCl_4	–	–	1000	1180	1	–	34.0
Berger and Walter [17]	Water	Toluene			998.2	866.7	1.003	0.586	35.4
		n-Butyl acetate				881.5		0.73	14.1
		n-Butanol				810		2.948	1.75

Pietzsch and Blass [31]	Water	TBP + *n*-alkane	–	–	1000.5	815.4	1.06	1.79	10.2
Kleczek et al. [32]	Water, glycerine	Six organics[a]	Seven organics[b]	c → d, d → c	1000–1054	860–1600	1–1.7	0.6–0.97	11.1–41.2
Lorenz et al. [18]	Water	Toluene, butyl acetate Butanol	–	–	998.2	866.7, 881.5 810	1.003	0.586, 0.73 2.948	35.4, 14.1 1.75
Bahmanyar et al. [53]	Water	Cumene	Acetic acid	d → c	998	862	0.998	0.78	29
Mohanty and Vogelpohl [37]	Water	Butyl acetate	–	–	998.2	881.5	1.003	0.73	14.1
Srinivasulu et al. [35]	Water	Kerosene	Butyric acid	d → c	998	800	1	1.67	44
Venkatnarasaiah and Verma [45]	Water	Kerosene	*n*-Butyric acid, benzoic acid	d → c	998	800	1.0	1.67	44
Luo et al. [36]	Water	(30%) TBP in kerosene	HNO_3	c → d	988	835	1.05	2.09	9.95
Vatanatham et al. [55]	Water	Toluene	Acetone	d → c	998	870	0.998	0.50	36.1
Li et al. [56]	Water	(30%) TBP in kerosene	HNO_3	c → d	–	–	–	–	–
He et al. [46]	Water	Benzene	Caprolactum	c → d	1060	890	–	–	–
Luo et al. [54]	Water + HNO_3	30% TBP in kerosene	TBP	d → c	988	835	1.05	2.09	9.95
Tang et al. [64]	Water	30% TBP in kerosene	HNO_3	d → c	998	835	1.05	2.09	9.95
Tribess and Brunello [19]	Water	Toluene	Acetone	d → c	998	864	1.0	0.585	34
Usman et al. [63]	Water	Acetic acid	Ethyl acetate	c → d	998	905	1.0	0.495	8.5

(1) The study [55] involve both MIBK and water as continuous phase.

[a] Benzene, toluene, xylene, butyl acetate, carbon tetrachloride and chloroform as the dispersed phase.

[b] Acetic, propionic, butyric and benzoic acids, acetone, phenol and iodine were used as solutes.

let line for the second phase. Different flow regimes in pulsed column have been plotted as a function of pulse velocity in Fig. 3a and b.

(a) *Flooding due to insufficient pulsing*: In the absence or low value of pulsation the drops coming from the sparger cannot pass through the plate holes of small diameter. As pulsed sieve plate columns do not have any downcomer, pulsing is the only source of energy for movement of the phases along the column height. Hence, if pulse velocity is small, it cannot pass the liquids alternately through the plate holes and flooding occurs. This condition is called as flooding due to insufficient pulsing. As pulse velocity ($A \times f$) is increased column capacity increases linearly with it, hence

$$(V_c + V_d)_f = A \times f \tag{1}$$

However, Berger et al. [6] have shown that the condition of no flow due to density difference occurs for systems of higher interfacial tensions. This is because large drops are formed for higher interfacial tension systems and plate holes can prevent them from moving to the next plate in absence of pulsation. Whereas drops formed for systems of low interfacial tension are small and they can pass through the plate holes even in absence of pulsation. Thus, for liquid–liquid systems with low interfacial tension throughput higher than Eq. (1) is obtained.

(b) *Mixer-settler regime*: In this regime (Fig. 2), the column behaves like a series of mixers and settlers. The regime is characterized by the separation of light and heavy phases into discrete, clear layers in the inter-plate spaces during the quiescent portion of the pulse cycle. Due to presence of a thick layer of the dispersed phase, hold-up is high in this case. As the pulse velocity (amplitude × frequency) is increased further, the large drops formed due to pulsations do not have sufficient time to collect under the plates and above mentioned distinct separation of the two phases is reduced. These large drops have low residence time. Therefore, hold-up decreases and reaches to a minimum value.

(c) *Dispersion regime*: The point of minimum hold-up is the beginning of the dispersion regime. This regime is characterized by non-uniform drop size distribution and no coalescence of dispersed phase drops. With increase in pulse velocity, drop size decreases and hold-up starts increasing in this regime.

(d) *Emulsion regime*: Further increase in pulse velocity, increases the inertial and shear forces on drops. These forces enhance drop breakage. Thus, an emulsion is formed. This regime occurs at high pulse velocity and there is no retardation of drops underneath sieve plates. Fine drops in the vicinity of the plates get carried away with the continuous phase through the plate holes during the reverse stroke of the pulsation. Hold-up increases very rapidly in this regime.

(e) *Unstable regime*: This regime is characterized by local phase inversion in different parts of the column. As the pulse velocity increases the shear on the dispersed phase drops increases. This results in decreased drop size which leads

Table 3
Experimental studies on pulsed sieve plate columns: details of parameters studied by different investigators

Investigators	Flow regimes/flooding	Drop size	Hold-up	Mass transfer
Cohen and Beyer [39]			√	√
Sege and Woodfield [3]				√
Chantry et al. [49]				√
Li and Newton [50]				√
Thornton [13]	√			√
Logsdail and Thornton [40]				√
Sobotic and Himmelblau [51]				√
Smoot and Babb [52]				√
Jones [22]		√		
Sehmel and Babb [5]			√	
Miyauchi and Oya [9]		√	√	
Kagan et al. [15]	√	√	√	
Angelino et al. [24]				√
Mishra and Dutt [41]			√	
Bell and Babb [42]			√	
Khemongkorn et al. [29]		√	√	√
Berger et al. [6]				√
Boyadzhiev and Spassov [10]		√		
Berger and Walter [17]	√			
Pietzsch and Blass [31]		√	√	
Kleczek et al. [32]		√		
Lorenz et al. [18]	√	√	√	
Bahmanyar et al. [53]				√
Mohanty and Vogelpohl [37]		√	√	
Srinivasulu et al. [35]		√		
Venkatnarasaiah and Verma [45]			√	
Luo et al. [36]				√
Vatanatham et al. [55]				√
Li et al. [56]				√
He et al. [46]				√
Luo et al. [53]		√		
Tang et al. [64]				
Tribess and Brunello [19]	√			
Usman et al. [63]				√

to a very large increase in hold-up. Any further increase in pulse velocity leads to flooding.

(f) *Flooding due to excessive pulsing*: With further increase in pulse velocity for a given phase flow rate, shear forces on the drops increase and fine drops are produced. These drops have a terminal rise velocity less than the superficial velocity of the continuous phase. Hence, they start accumulating in the disengaging section of the column at the continuous phase outlet. At higher pulse velocity drops start going through the continuous phase outlet. This is called as flooding due to excessive pulsing.

Since the flow conditions in the column differ according to the regimes of operations, it is important to know where a particular regime begins. The transition from one type of regime to another is a function of changes in pulsing conditions or throughput for a given liquid–liquid system and column geometry. Since these conditions change for different investigators, there are a large number of correlations for regime transition as listed by Kumar and Hartland [7]. Major correlations for regime transition have been listed in Table 4.

Sehmel and Babb [5] in their study on hold-up in pulsed column observed that it passes through a minimum with increase in frequency. They reported that the frequency corresponding to the minimum hold-up was also the frequency which marked the visually observed transition from mixer-settler to dispersion regime. The transition frequency was correlated as a function of physical properties and pulse amplitude as,

$$f_{\mathrm{t}} = 0.667(-3.373 + 3.883 \times 10^{2}\mu_{\mathrm{d}}\sigma\Delta\rho - \ln A) \tag{2}$$

Working with the water–acetic acid–MIBK, Sato et al. [8] gave three different correlations (Table 4) for transition from mixer-settler to emulsion regime. Boyadzhiev and Spassov [10] formulated a criterion for intensive pulsation by using the experimental data of eight different investigators. It can be used for excluding the data points in mixer-settler and dispersion regime from emulsion regime. Kumar and Hartland [11], and Tung and Leucke [12] have used the experimental observation on hold-up of different investigators to formulate regime transition criteria between dispersion and emulsion regime. Kumar and Hartland [7] have suggested the following criterion by taking experimental data from 15 different investigators.

$$(Af)_{\mathrm{t}} = 9.69 \times 10^{-3}\left(\frac{\sigma\Delta\rho^{1/4}\alpha}{\mu_{\mathrm{d}}^{3/4}}\right)^{0.33} \tag{3}$$

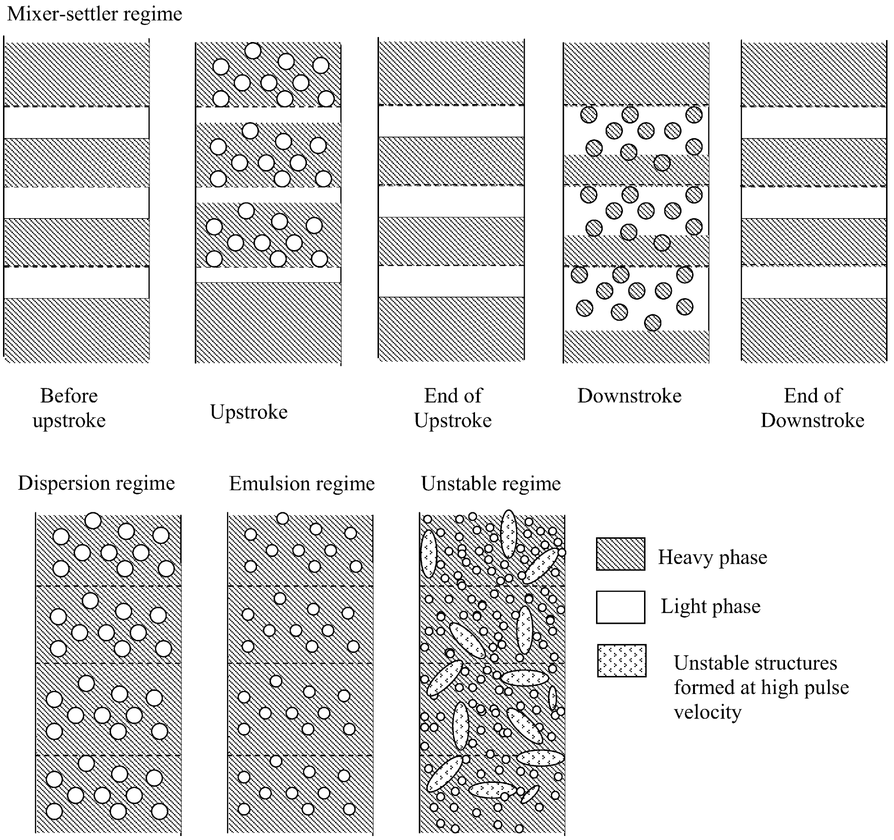

Fig. 2. Flow regimes in pulsed sieve plate columns.

It can be used for excluding the data points in the mixer-settler regime ($(Af) \leq (Af)_t$) from the dispersion and emulsion regime ($(Af) > (Af)_t$). Applicability of the correlations of Sehmel and Babb [5], Sato et al. [8] or Miyauchi and Oya [9] were found to be limited to the liquid–liquid systems and column geometry for which they were formulated. Whereas the correlations of Kumar and Hartland [7] and Boyadzhiev and Spassov [10], have been formulated over a large data base. Hence, they were used for regime classification of the experimental data gathered from the work of different investigators.

2.2. Flooding characteristics of pulsed sieve plate columns

Thornton [13] considered the pulsed column to behave like spray column under high pulsations. He differentiated the slip velocity (V_{slip}) relation (Eq. (4)) to obtain the flooding capacity with respect to continuous and dispersed phase velocities.

$$\frac{V_d}{\phi} + \frac{V_c}{(1-\phi)} = V_0(1-\phi) \tag{4}$$

Using the experimental data obtained on five liquid–liquid systems, he gave a correlation for predicting the characteristic velocity (V_0)

$$\frac{V_0\mu_c}{\sigma} = 0.60\left(\frac{\varepsilon\mu_c^5}{\rho_c\sigma^4}\right)^{-0.24}\left(\frac{d\rho_c\sigma}{\mu_c^2}\right)^{0.90}\left(\frac{\mu_c^4 g}{\Delta\rho\sigma^3}\right)^{1.01} \times\left(\frac{\Delta\rho}{\rho_c}\right)^{1.80}\left(\frac{\mu_d}{\mu_c}\right)^{0.30} \quad \text{for } Af > 0.012\,\text{m/s} \tag{5}$$

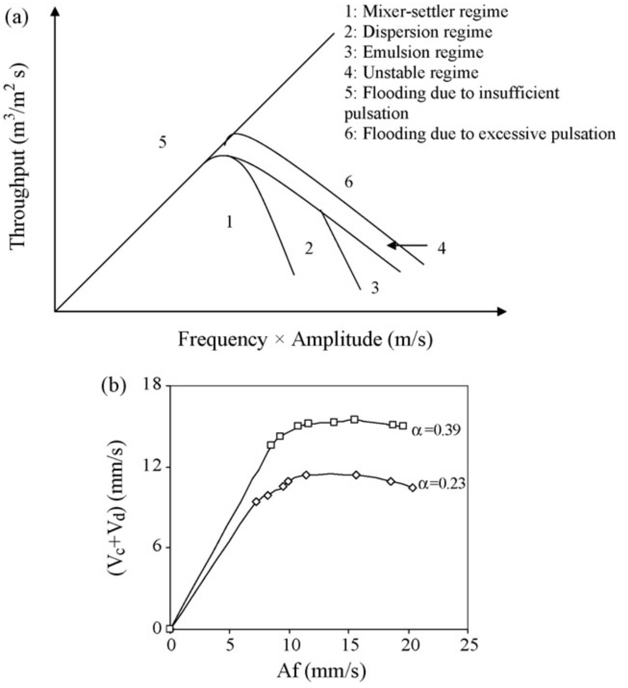

Fig. 3. Flow regimes in pulsed sieve plate extraction column. (a) Different flow regimes in pulsed sieve plate columns and (b) flooding curve as predicted by Lorenz et al. [18] for $\alpha = 0.23$, 0.39.

Table 4
Correlations for predicting regime transition in pulsed sieve plate columns

Investigators	Correlation for regime transition	Remark
Sehmel and Babb [5]	$f_t = 0.667(-3.373 + 3.883 \times 10^2 \mu_d \sigma \Delta\rho - \ln A)$	$f < f_t$ Mixer-settler regime $f \geq f_t$ Dispersion regime
Sato et al. [8]	$0 < Af \leq 1.3 V_d^{0.22} h^{-0.32} d^{-0.37}$	Mixer-settler regime
	$V_d^{0.22} h^{-0.32} d^{-0.37} < Af \leq 1.3 V_d^{0.22} h^{-0.35} d^{-0.37}$	Dispersion regime
	$Af > 0.8 V_d^{0.22} h^{-0.35} d^{-0.37}$	Emulsion regime
Miyauchi and Oya [9]	$Af < 0.0021(\beta h)^{1/3} \left(\frac{\mu_d^2}{\sigma \Delta\rho}\right)^{-1/4}$	Dispersion regime
	$Af > 0.0021(\beta h)^{1/3} \left(\frac{\mu_d^2}{\sigma \Delta\rho}\right)^{-1/4}, \quad \beta = \frac{\alpha^2}{(1-\alpha)(1-\alpha^2)}$	Emulsion regime
Boyadzhiev and Spassov [10]	$Af < 0.5 \left(\frac{0.96\alpha^2}{\rho_c}\right)^{1/3}$	Dispersion regime
	$Af \geq 0.5 \left(\frac{0.96\alpha^2}{\rho_c}\right)^{1/3}$	Emulsion regime
Kumar and Hartland [11]	$Af < \left[0.05 \beta h \left(\frac{\rho_c}{\Delta\rho^{3/4} \sigma^{1/4} g^{5/4}}\right)^{-1}\right]^{1/3}$	Dispersion regime
	$Af \geq \left[0.05 \beta h \left(\frac{\rho_c}{\Delta\rho^{3/4} \sigma^{1/4} g^{5/4}}\right)^{-1}\right]^{1/3}$	Emulsion regime
Tung and Luecke [12]	$Af < \left(\frac{0.062\alpha^2}{\rho_c}\right)^{1/3}$	Dispersion regime
	$Af \geq \left(\frac{0.062\alpha^2}{\rho_c}\right)^{1/3}$	Emulsion regime
Kumar and Hartland [7]	$(Af)_t = 9.69 \times 10^{-3} \left(\frac{\sigma \Delta\rho^{1/4} \alpha}{\mu_d^{3/4}}\right)^{0.33}$	$Af < (Af)_t$ Mixer-settler regime $Af \geq (Af)_t$ Dispersion regime

where ε in Eq. (5) is the power dissipated per unit mass of fluid and is calculated by using

$$\varepsilon = \frac{\pi^2(1-\alpha^2)}{2\alpha^2 c_o^2 h}(Af)^3 \tag{6}$$

Eq. (5) has been developed using water as the continuous phase hence it does not include variation in the physical properties of this phase. Flooding correlation of this author can be put as:

$$\begin{aligned}(V_c + V_d)_f = {} & 0.60\sigma^{-0.17} \mu_c^{-0.26} K^{-0.24} A^{-0.72} f^{-0.72} h^{0.24} \\ & \times \rho_c^{-0.66} d^{0.9} \Delta\rho^{0.79} g^{1.01} \mu_d^{0.30} ((1 - 2\phi_f) \\ & \times (1 - \phi_f)^2 + \phi_f^2 (1 - \phi_f))\end{aligned} \tag{7}$$

$$K = \left(\frac{\pi^2}{2C_0^2}\right)\left(\frac{1-\alpha^2}{\alpha^2}\right)$$

$$\phi_f = \frac{\sqrt{R^2 + 8R} - 3R}{4(1-R)}, \quad R = \left(\frac{V_d}{V_c}\right)_f \tag{8}$$

The flooding correlation of Thornton [13] considers hold-up at flooding (ϕ_f) as a function of flow ratio of the two phases only. Thus, it does not take into account the effect of the physical properties of the phases and geometrical and operating parameters of the column on ϕ_f. However, the flooding velocities incorporate the effect of all these parameters through the characteristic velocity (V_0).

Smoot et al. [4] combined the experimental data of different investigators and proposed a correlation for emulsion flooding.

$$\begin{aligned}(V_c + V_d)_f = {} & 0.527(\Delta\rho)^{0.63} \rho_c^{-0.775} \mu_c^{0.489} \mu_d^{-0.20} g^{0.81} V_c^{-0.014} \\ & \times V_d^{0.014} h^{0.207} d^{0.458} K^{-0.207} A^{-0.621} f^{-0.621}\end{aligned} \tag{9}$$

However, McAllister et al. [14] have shown that in spite of the fact that the correlation of Smoot et al. [4] (Eq. (9) appears to be quite different from Eq. (7); both give very similar curves of calculated $(V_c + V_d)_f$ versus Af. From Eqs. (7) and (9), we can observe that the exponent on Af for Thronton [13] is −0.72 and for Smoot et al. [4] the exponent is −0.621. Further observations for exponent on other variables give the similarity between the two correlations. Furthermore, Smoot et al. [4] have not given the criteria to classify the regime of the experimental data gathered by them.

Kagan et al. [15] studied flooding behaviour of a 0.056 m diameter column for water–kerosene system. They have varied the kerosene to water flow ratio from 1:2 to 1:10 and found that the column reached its maximum capacity at pulse velocity of 0.01 m/s. The maximum column capacity was found to decrease with increase in phase flow ratio (V_d/V_c). They also conducted an investigation of the individual effect of frequency and amplitude and found that the product *fA* can be used instead of *f* or *A* separately for describing the behaviour of the column. They proposed the following correlation using regression analysis.

For ascending part (Fig. 3) of the $(V_c + V_d)_f$ versus *Af* curve

$$V_{df} = 2.5 \times 10^{-2} V_c^{-1.1} V_d^{1.1} D^{-0.69} g^{-0.19} A^{1.0} f^{1.62} h^{0.5} \qquad (10)$$

For descending part (Fig. 3) of the $(V_c + V_d)_f$ versus *Af* curve

$$V_{df} = 3.0 \times 10^{-9} V_c^{-1.1} V_d^{1.1} D^{0.95} g^{-0.3} A^{-1.75} f^{-2.6} h^{0.5} \qquad (11)$$

The positive exponents on *A* and *f* in Eq. (10) show that in mixer-settler regime as pulse velocity increases capacity of the column increases and flooding occurs at higher flow rates. The negative exponents on *A* and *f* in Eq. (11) indicate that once the column reaches its maximum capacity (Fig. 3) further increase in pulse velocity decreases its capacity. In view of the fact that the experiments were carried out only on kerosene–water system, they excluded the terms, which account for influence of variation of physicochemical properties of the liquid–liquid systems. This limits the applicability of their correlation to liquid–liquid systems having physical properties similar to kerosene–water.

McAllister et al. [14] formulated a correlation for flooding by taking a large amount of data of earlier investigators.

$$\begin{aligned}\ln\left(\frac{V_c + V_d}{\varDelta_c + \varDelta_d}\right) = {} & -3.741 + 0.2568\ln X - 0.07194(\ln X)^2 \\ & + 0.006191(\ln X)^3 - 1.034\ln Y \\ & - 0.09096(\ln Y)^2 - 0.0008898(\ln Y)^3 \\ & + 0.1424\ \ln\left(\frac{V_c}{V_d}\right) \\ & - 0.1807\ln\left(\frac{A}{h}\right) + 0.07198\ln\left(\frac{X}{Y}\right),\end{aligned}$$

$$X = \frac{\Delta\rho d\alpha^2 g}{\rho_c \pi_M^2};$$

$$Y = \frac{\mu_c \pi_v}{\alpha\sigma} \varDelta_c = Af\left[\sqrt{1-\lambda^2} + \lambda\left(\frac{\pi}{2} + \sin^{-1}\lambda\right)\right],$$

$$\varDelta_d = Af\left[\sqrt{1-\lambda^2} - \lambda\left(\frac{\pi}{2} - \sin^{-1}\lambda\right)\right] \lambda = \frac{V_{cf} - V_{df}}{\pi Af} \qquad (12)$$

Physical significance of the different terms in Eq. (12) is given in a report by Groenier et al. [16].

Berger and Walter [17] measured flooding for different systems and geometrical parameters and found that if the phase ratio V_d/V_c is increased, the maximum of the flooding curves decreased and the new maximum was found to occur at smaller frequencies (Fig. 3), i.e. column was approaching flooding at a lower frequency. This could be because of increase in the dispersed phase flow rate, increases the resistance to the flow of continuous phase along the column. They established an interrelationship between the frequency at which the maximum in the flooding curve occurred and the phase flow ratio for all three types of liquid–liquid systems studied by them.

With a smaller interfacial tension, smaller drops are formed which can readily pass through holes. This shows that the maximum frequency (f_m) at which flooding occurs is a function of interfacial tension besides the phase ratio. The fractional free area of the plate was found to have no influence on f_m. They compared the correlations of Smoot et al. [4] and McAllister et al. [14] with their experimental data and found that these correlations though contain physical properties data, the phase ratio, pulsation data and plate geometry; they do not predict the shape of the flooding curve. Even for emulsion regime they gave values which were consistently higher. They corrected the original correlation of McAllister et al. [14] and observed that it can be applied only for very small hole diameter such that there is no flow due to density difference. Berger and Walter [17] proposed empirical correlations for calculating throughput at flooding and the frequency at which this occurs.

$$\begin{aligned}(V_c + V_d)_m = {} & (6.81 \times 10^{-3} + 0.7047\sigma - 15.222\sigma^2) \\ & \times (1 - 4.55\alpha + 3.247\alpha^2)1 + 0.1778\left(\ln\frac{V_c}{V_d}\right) \\ & + 0.0437\left(\ln\frac{V_c}{V_d}\right)^2 \qquad (13)\end{aligned}$$

$$\begin{aligned}f_m = {} & 0.4908 + 1.11 \times 10^2\sigma - 1.80 \times 10^3\sigma^2 \\ & - (3.44 \times 10^{-2} + 7.1\sigma)\ln\frac{V_d}{V_c} \qquad (14)\end{aligned}$$

The above correlation does not take into account the effect of mass transfer and plate spacing on flooding behaviour of the pulsed columns.

Lorenz et al. [18] measured flooding curves for three different systems with varying physical properties and it was found to exhibit the well-known parabolic behaviour with maxima at the transition from mixer-settler to dispersion regime.

Tribess and Brunello [19] showed that for the toluene–water system, maximum throughputs increased with plate spacing. It was due to fact that the shear forces on drops reduce with an increase in plate spacing which results in an increase in mean drop diameter when there are fewer plates. They also studied the effect of mass transfer on the flooding behaviour of pulsed columns for the system toluene–acetone–water. Mass transfer in this case was from dispersed phase to the continuous phase (d → c), i.e. from toluene to water. The maximum flow rate for column flooding was observed to increase in the presence of mass transfer. Thus, flooding in the column was delayed. This was because of formation of large drops due to Marangoni effect. These authors' added terms to the equation of Berger and Walter [17] to take into account the effect of mass transfer and plate spacing.

$$\begin{aligned}(V_c + V_d)_m = {} & (6.81 \times 10^{-3} + 0.7047\sigma - 15.222\sigma^2) \\ & \times (1 - 4.55\alpha + 3.247\alpha^2)1 + 0.1778\left(\ln\frac{V_c}{V_d}\right) \\ & + 0.0437\left(\ln\frac{V_c}{V_d}\right)^2 (0.2115D^{0.20}h^{0.18}) \\ & \times\left[1 + \left(\frac{V_c}{V_d}\frac{h}{d}C_t\right)\right]^{0.09} \qquad (15)\end{aligned}$$

$$f_{\mathrm{m}} = 0.4908 + 1.11 \times 10^2\sigma - 1.80 \times 10^3\sigma^2 - (3.44 \times 10^{-2} + 7.1\sigma)\ln\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}(2.464A^{-1}D^{0.20}h^{-0.01}) \times \left[1 + \left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\frac{h}{d}C_{\mathrm{t}}\right)\right]^{0.07} \quad (16)$$

where C_{t} is weight fraction of solute in the feed at inlet.

Different correlations for predicting flooding in pulsed columns have been listed in Table 5.

2.2.1. *Recommended correlation for scale-up*

To summarize, the flooding in the pulsed column is a function of different parameters. An increase in the plate parameters, e.g. hole diameter (d), percentage free area (α) or plate spacing (h) delay the flooding in the column and increase the throughput. However, it reduces the shear forces on drops which affect the mass transfer efficiency of the column. Increase in the pulse velocity ($A \times f$) has been found to increase the approach to flooding. Mass transfer from dispersed to continuous phase (d → c) gives larger drops due to Marangoni effect. It reduces the column hold-up. The reduction in hold-up delays the flooding tendency

Table 5
Correlations for predicting flooding in pulsed sieve plate columns

Investigators	Correlations	Remark
Thornton [13]	$V_{\mathrm{cf}} = V_0(1-2\phi_{\mathrm{f}})(1-\phi_{\mathrm{f}})^2, \quad V_{\mathrm{df}} = 2V_0\phi_{\mathrm{f}}^2(1-\phi_{\mathrm{f}})$	Can be used for calculating emulsion flooding
Smoot et al. [4]	$\frac{(V_{\mathrm{cf}}+V_{\mathrm{df}})\mu_{\mathrm{c}}}{\sigma} = 0.527\left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{-0.014}\left(\frac{\Delta\rho}{\rho_{\mathrm{c}}}\right)^{0.63}\left(\frac{\varepsilon_{\mathrm{m}}\mu_{\mathrm{c}}^5}{\rho_{\mathrm{c}}\sigma^4}\right)^{-0.207}\left(\frac{\mathrm{d}\sigma\rho_{\mathrm{c}}}{\mu_{\mathrm{c}}^2}\right)^{0.458}\left(\frac{D}{d}\right)^{0.0}\left(\frac{g\mu_{\mathrm{c}}^4}{\rho_{\mathrm{c}}\sigma^3}\right)^{0.81}\left(\frac{\mu_{\mathrm{d}}}{\mu_{\mathrm{c}}}\right)^{-0.20}$	Can be used for calculating emulsion flooding
McAllister et al. [14]	$\ln\left(\frac{V_{\mathrm{c}}+V_{\mathrm{d}}}{\Delta_{\mathrm{c}}+\Delta_{\mathrm{d}}}\right) = -3.741 + 0.2568\ln X - 0.07194(\ln X)^2 + 0.006191(\ln X)^3 - 1.034\ln Y - 0.09096(\ln Y)^2 - 0.0008898(\ln Y)^3 + 0.1424\ln\left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right) - 0.1807\ln\left(\frac{A}{h}\right) + 0.07198\ln\left(\frac{X}{Y}\right)$ $X = \frac{\Delta\rho \mathrm{d}\alpha^2 g}{\rho_{\mathrm{c}}\pi_{\mathrm{M}}^2}; \quad Y = \frac{\mu_{\mathrm{c}}\pi_{\mathrm{v}}}{\alpha\sigma}\, \Delta_{\mathrm{c}} = Af\left[\sqrt{1-\lambda^2} + \lambda\left(\frac{\pi}{2} + \arcsin\lambda\right)\right]\, \Delta_{\mathrm{d}} = Af\left[\sqrt{1-\lambda^2} - \lambda\left(\frac{\pi}{2} - \arcsin\lambda\right)\right]\, \lambda = \frac{V_{\mathrm{cf}}-V_{\mathrm{df}}}{\pi Af}$	Applicable only for small hole diameter such that there is no flow due to density difference
Kagan et al. [15]	$\frac{V_{\mathrm{d}}}{gD} = 2.5 \times 10^{-2}\left(\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}\right)^{1.1}\left(\frac{A}{D}\right)^{1.0}\left(\frac{f^2D}{g}\right)^{0.81}\left(\frac{h}{D}\right)^{0.5}$	For ascending part of flooding curve
	$\frac{V_{\mathrm{df}}}{gD} = 3.0 \times 10^{-9}\left(\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}\right)^{1.1}\left(\frac{A}{D}\right)^{-1.75}\left(\frac{f^2D}{g}\right)^{-1.3}\left(\frac{h}{D}\right)^{0.5}$	For descending part of flooding curve
Berger and Walter [17]	$(V_{\mathrm{cf}}+V_{\mathrm{df}})_{\mathrm{m}} = (6.81 \times 10^{-3} + 0.7047\sigma - 15.222\sigma^2)(1 - 4.55\alpha + 3.247\alpha^2)1 + 0.1778\left(\ln\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right) + 0.0437\left(\ln\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^2$ $f_{\mathrm{m}} = 0.4908 + 1.11 \times 10^2\sigma - 1.80 \times 10^3\sigma^2 - (3.44 \times 10^{-2} + 7.1\sigma)\ln\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}$	Studies have been done in absence of mass transfer
Tribess and Brunello [19]	$(V_{\mathrm{cf}}+V_{\mathrm{df}})_{\mathrm{m}} = (6.81 \times 10^{-3} + 0.7047\sigma - 15.222\sigma^2)(1 - 4.55\alpha + 3.247\alpha^2)1 + 0.1778\left(\ln\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right) + 0.0437\left(\ln\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^2(0.2115D^{0.20}h^{0.18})\left[1 + \left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\frac{h}{d}C_{\mathrm{t}}\right)\right]^{0.09}$	Studies have been done in presence of mass transfer in d → c direction.
	$f_{\mathrm{m}} = 0.4908 + 1.11 \times 10^2\sigma - 1.80 \times 10^3\sigma^2 - (3.44 \times 10^{-2} + 7.1\sigma)\ln\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}(2.464A^{-1}D^{0.20}h^{-0.01})\left[1 + \left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\frac{h}{d}C_{\mathrm{t}}\right)\right]^{0.07}$	Column diameter was very small (D = 0.0394 m)

Table 6
Variation in the experimental data used for testing different correlation

Parameter	Flooding	Drop size	Hold-up	Mass transfer
$D \times 10^{-3}$ (m)	39.6–80	25–225	43–225	25.4–600
$d \times 10^{-3}$ (m)	2–6.5	2–6	2–8	1–8
α (%)	23–60	18–49	23–49	9–46
$h \times 10^{-3}$ (m)	50–100	50–100	50–100	38.1–100
$fA \times 10^{-3}$ (m/s)	0–52.85	2.46–78.20	2.45–84.50	2.25–125
$V_c \times 10^{-3}$ (m/s)	0.3–20.4	1.36–6.00	0–7.2	0.2–8.0
$V_d \times 10^{-3}$ (m/s)	0.7–14.3	0.49–6.10	1.05–6.10	0.3–8.0
ρ_c (kg/m^3)	990–1000	998–1000	998–1000	691–1060
ρ_d (kg/m^3)	810–881.5	798–1590	660–958.4	691–1436
$\mu_c \times 10^{-3}$ (Pa s)	1.00–1.03	1.00–1.05	1.00–1.03	0.5–1.1
$\mu_d \times 10^{-3}$ (Pa s)	0.58–3.6	0.59–2.95	0.29–2.95	0.5–4.88
$\sigma \times 10^{-3}$ (N/m)	1.75–36.00	1.75–45.00	1.75–46.50	4.6–44.0
$D \times 10^{-3}$ (m)	39.6–80	25–225	43–225	25.4–600
$d \times 10^{-3}$ (m)	2–6.5	2–6	2–8	1–8
α (%)	23–60	18–49	23–49	9–46
$h \times 10^{-3}$ (m)	50–100	50–100	50–100	38.1–100
$fA \times 10^{-3}$ (m/s)	0–52.85	2.46–78.20	2.45–84.50	2.25–125
$V_c \times 10^{-3}$ (m/s)	0.3–20.4	1.36–6.00	0–7.2	0.2–8.0
$V_d \times 10^{-3}$ (m/s)	0.7–14.3	0.49–6.10	1.05–6.10	0.3–8.0
ρ_c (kg/m^3)	990–1000	998–1000	998–1000	691–1060
ρ_d (kg/m^3)	810–881.5	798–1590	660–958.4	691–1436
$\mu_c \times 10^{-3}$ (Pa s)	1.00–1.03	1.00–1.05	1.00–1.03	0.5–1.1
$\mu_d \times 10^{-3}$ (Pa s)	0.58–3.6	0.59–2.95	0.29–2.95	0.5–4.88
$\sigma \times 10^{-3}$ (N/m)	1.75–36.00	1.75–45.00	1.75–46.50	4.6–44.0

of the column and thus increases throughput as discussed by Tribess and Brunello [19].

A total of 400 experimental data points from the investigation of Berger et al. [6], Berger and Walter [17], Lorenz et al. [18] and Tribess and Brunello [19] were taken for testing the correlations on flooding. Table 6 shows the variation in the experimental data with respect to the geometrical and operating parameters and physical properties.

Two approaches viz. predicting the entire flooding curve [4,13,14] and predicting only the maximum in the flooding curve [17,19] have been reported in the literature. Both these approaches were used for the gathered data set. Fig. 4a and b show the predictive ability of the correlations proposed by Smoot et al. [4] and McAllister et al. [14], respectively. Poor predictive ability of Smoot et al. [4] correlation can be due to the small variation in physical properties, geometrical and operating parameters of the data set over which it was formulated. McAllister et al. [14] correlation, which is formulated over the largest data base is not applicable for large hole diameters and small interfacial liquid–liquid systems [17]. Hence, it can be concluded that none of the correlations available in the literature are general enough to predict the entire flooding curve for a liquid–liquid system. Thus, a comprehensive experimental work is still required. However, as pointed out by Lorenz et al. [18], the cost of these tests can be reduced if data on fluid dynamic and mass transfer are known for the three liquid–liquid systems (*n*-butanol–succinic acid–water, *n*-butyl acetate–acetone–water and toluene–acetone–water) as proposed by the European Federation of Chemical Engineering, EFCE (Misek et al. [20]). As these systems have very different interfacial tensions, they permit behavior prediction of extraction columns for other systems. The experimental data gathered by us on flooding are primarily for these systems.

From design point of view (for calculating column diameter) what is required is the maximum $(V_c + V_d)_m$ in the flooding curve. A total of 64 values of $(V_c + V_d)_m$ were obtained from the 400 data points. The predictive ability of the two correlations proposed by Berger and Walter [17] and Tribess and Brunello [19] is shown in Fig. 4c and d, respectively. The correlation of Tribess and Brunello [19] includes the effect of mass transfer on flooding as compared to Berger and Walter [17] correlation. Also the latter correlation [17] does not take into account the effect of plate spacing. Hence, Tribess and Brunello [19] correlation is recommended for predicting the maximum in the flooding curve.

2.3. Drop size in pulsed sieve plate column

At low amplitude and frequency of pulsation, the break-up of drops is controlled by the ratio of buoyancy and interfacial tension forces. Thus, the following equation may be used to calculate the maximum drop size to be expected.

$$d_{\max} = C_1 \left(\frac{\sigma}{\Delta\rho g} \right)^{0.5} \tag{17}$$

The constant C_1 is a function of column geometry. Logsdail and Slater [21] have suggested a value of 0.92 for pulsed sieve plate column. Since the above equation does not include the

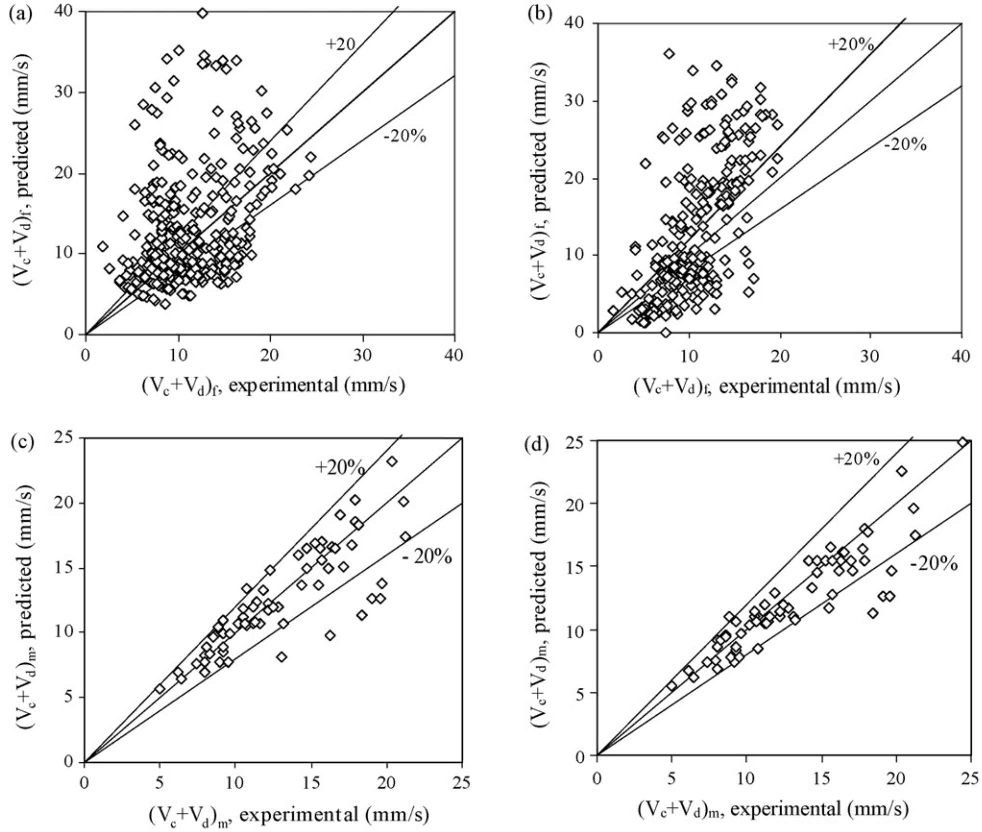

Fig. 4. Testing the predictive ability of different correlations for flooding in pulsed sieve plate columns: (a) Smoot et al. [4], (b) McAllister et al. [14], (c) Berger and Walter [17], and (d) Tribess and Brunello [19].

effect of shear forces on drops, it is not valid if the drop sizes are similar to plate hole sizes.

Jones [22] measured drop size in a pulsed column of 0.0254 m diameter using water–MIBK system. They observed that multi-sized drops are formed at lower pulse velocity whereas at high pulse velocities more uniform drops are produced. They suggested that the product of amplitude and frequency (*Af*) control the drop size produced in the column. In his studies, the drop size along the column height changed very little from the bottom-most observation point to top of the column. The majority of drop breakage was observed to occur during passage through the first two or three sieve plates. He observed that the flow rates of either phase have very little effect on the size of drops. Larger drops were observed with large hole diameter. They presented a graphical correlation for predicting the d_{32} as a function of $\sqrt{d}/V_a$ with d as hole diameter and V_a as mean orifice velocity calculated as follows.

$$V_a = \frac{2fA}{\alpha} + \frac{1}{2\alpha A_c}(Q_d - Q_c) \tag{18}$$

Misek [23] proposed the following empirical correlation for predicting drop size in pulsed columns. In addition to physical properties and operating parameters, d_{32} was found to be proportional to hole diameter, $d^{0.4}$.

$$d_{32} = 0.439\sigma^{0.6}\rho_c^{-0.6}\alpha^{0.3}d^{0.4}(Af + V_c)^{-1.2} \tag{19}$$

Miyauchi and Oya [9] measured drop size for water–MIBK system in absence of mass transfer in a column of 0.054 m diameter. Drop size was observed to increase with an increase in plate spacing. With increase in plate spacing, the residence time of drops between two plates increase and this enhances probability for drop coalescence, leading to large drops. Also if plate spacing is increased at a given pulse velocity the frequency of application of shear forces on the drops decreases hence drop size increases. For fixed velocity of MIBK at 0.43 mm/s their data is well correlated by the empirical formula

$$d_{32} = 2.03 \times 10^{-5}A^{-1.2}f^{-1.2}h^{0.4} \quad \text{for} \quad \frac{Af}{h^{1/3}} > 5.57 \times 10^{-2}\,\text{m}^{2/3}/\text{s} \tag{20}$$

However, for $Af/h^{1/3} < 5.57 \times 10^{-2}$ m$^{2/3}$/s, d_{32} was found to deviate gradually from the given correlation, with exponent on *Af* changing from −1.2 to −0.6. Also the above correlation is developed for very low velocity of the dispersed phase. This limits the applicability of this correlation for higher V_d.

Kagan et al. [15] studied drop size in pulsed column of 0.056 m diameter for the system kerosene–water. With an increase in the pulse velocity, the drop size along the column height was observed to decrease. The perforation diameter in their experiment was 2 mm. Thus, even though column active height was 4 m, the limit for droplet break-up in the turbulent stream was not achieved resulting in smallest drops at top. They also showed that varying the kerosene to water ratio from 0.1

to 0.5 had no influence on the droplet size. They proposed the following empirical correlation for their experimental data.

$$d_{32} = 0.92\sigma^{0.5}\mu_{\mathrm{c}}^{0.1}\rho_{\mathrm{c}}^{-0.6}g^{-0.4}A^{-0.3}f^{-0.3} \tag{21}$$

Angelino et al. [24], Pilhofer [25] and Ugarcic [26] cited from Kumar and Hartland [27] proposed correlations of the following form for predicting drop size.

$$d_{32} = C\left(\frac{\sigma}{\rho_{\mathrm{c}}}\right)^{0.6}(Af)^{-1.2} \tag{22}$$

Correlations of these investigators are based on the Kolmogorov's theory of isotropic turbulence. According to this theory, $d\alpha(\sigma/\rho_{\mathrm{c}})^{0.6}\varepsilon^{-0.4}$.

Two correlations are available for calculating ε. Jealous and Johnson [28] do not consider the pressure recovery for fluid after passing through the plate holes and gave the power dissipation per unit mass as

$$\varepsilon = \frac{\pi^2(1-\alpha^2)}{2\alpha^2 c_0^2 h}(Af)^3 \tag{23}$$

On the other hand, Miyauchi and Oya [9] considered pressure recovery after the fluid passes through plate holes. This resulted in

$$\varepsilon = \frac{5\pi^2(1-\alpha)(1-\alpha^2)}{6\sqrt{2}\alpha^2 c_0^2 h}(Af)^3 \tag{24}$$

Since Eq. (24) considers the pressure recovery for the fluid once it passes through the plate holes, it gives a closer picture of the flow around such holes. Hence, it is recommended for calculating the energy dissipation per unit mass for pulsed sieve plate column.

Since both the above correlations shows $\varepsilon\alpha(Af)^3$, this results in $d\alpha(Af)^{-1.2}$. Because of large exponent on Af, correlations based on Kolmogorov's theory of isotropic turbulence exaggerate the influence of pulsation intensity on the drop size and yield excessively large drop diameters at moderate levels of pulsations (especially in mixer-settler regime). Hence, correlations based on the theory of isotropic turbulence cannot be applied for the entire operating range of the pulsed column.

Khemogkorn et al. [29] studied the effect of mass transfer direction on drop size using water–iodine–CCl_4. Drop size was observed to reduce as A or f increased, and different influence of A and f were detected. They observed smaller drop sizes for mass transfer from dispersed to continuous phase (d → c) than from continuous to dispersed phase (c → d). Influence of the dispersed phase flow rate on drop size was observed to be very small. Decrease in hole diameter was observed to result in smaller drops due to increase in shear forces on the them. They proposed two correlations for calculating d_{32} (in dispersion or emulsion regime) depending on the direction of mass transfer.

$$d_{32} = 2.59\times10^{-5}A^{-1.0}f^{-1.24}N^{-0.21}Vd^{-0.083} \quad \text{for} \quad \mathrm{c}\rightarrow\mathrm{d} \tag{25}$$

$$d_{32} = 4.90\times10^{-5}A^{-1.0}f^{-1.24}N^{-0.21}Vd^{-0.0002} \quad \text{for} \quad \mathrm{d}\rightarrow\mathrm{c} \tag{26}$$

Boyadzhiev and Spassov [10] measured drop size in a column of 0.05 m diameter. Water was the continuous phase, while the dispersed phase was a mixture of kerosene and CCl_4. They observed uniform drop size in the top portion of the column. For the effect of phase flow rates they reported that if $V_{\mathrm{c}} + V_{\mathrm{d}} \ll Af$, then phase flow rates do not influence the mean droplet diameter, primarily because pulse velocity ≫ phase velocity under such conditions. By taking experimental data of other investigators they proposed the following correlation for calculation of drop size in emulsion regime of operation. Thus, drop size was found to be proportional to $d^{0.4}$ even in the emulsion regime.

$$d_{32} = 0.25\sigma^{0.6}\rho_{\mathrm{c}}^{-0.6}\alpha^{0.8}d^{0.4}A^{-1.2}f^{-1.2} \quad \text{for} \quad \frac{\rho_{\mathrm{c}}(2Af)^3}{(2\alpha^2)} > 0.48\,\mathrm{kg/s^3} \tag{27}$$

Pietzsch and Pilhofer [30] formulated the force balance model for predicting drop size in pulsed columns. They equated the buoyancy, inertial and drag forces which cause the drop breakup to the interfacial tension forces which stabilize a drop.

$$\begin{aligned}&\text{buoyancy force}: F_{\mathrm{A}} = \left(\frac{\pi}{6}\right)d_{\mathrm{p}}^3\Delta\rho g,\\ &\text{inertial force}: F_{\mathrm{w}} = \left(\frac{\pi}{6}\right)d_{\mathrm{p}}^3\rho_{\mathrm{d}}b\\ &\text{drag force}: F_{\mathrm{D}} = \left(\frac{c_{\mathrm{D}}\rho_{\mathrm{c}}V_{\mathrm{m}}^2}{2}\right)\left(\frac{\pi}{4}d_{\mathrm{p}}^2\right),\\ &\text{interfacial tension force}: F_{\mathrm{I}} = \pi\sigma d_{\mathrm{p}}\end{aligned} \tag{28}$$

Here, 'b' is the deceleration suffered by drop once it crosses the vena contracta. The first three forces tend to break the drop; the last one stabilizes a drop. Consequently, if there is a balance of the forces, a stable drop exists with diameter d_{p}. The balance of above forces leads to the following quadratic equation.

$$\left(\frac{\Delta\rho g + b\rho_{\mathrm{d}}}{6\sigma}\right)d_{\mathrm{p}}^2 + \left(\frac{c_{\mathrm{w}}V_{\mathrm{m}}^2\rho_{\mathrm{c}}}{8\sigma}\right)d_{\mathrm{p}} - 1 = 0$$

The solution of the above equation gives,

$$d_{\mathrm{p}} = \sqrt{\frac{6\sigma}{\Delta\rho g + b\rho_{\mathrm{d}}} + \frac{9}{64}\left(\frac{c_{\mathrm{D}}V_{\mathrm{m}}^2\rho_{\mathrm{c}}}{\Delta\rho g + b\rho_{\mathrm{d}}}\right)^2} - \frac{3}{8}\frac{c_{\mathrm{D}}V_{\mathrm{m}}^2\rho_{\mathrm{c}}}{\Delta\rho g + b\rho_{\mathrm{d}}} \tag{29}$$

The calculation of the drop size according to above equation requires the knowledge of the velocity of a drop V_{m} in the region of high velocity, the deceleration 'b', and the drag coefficient C_{D}. V_{m} is calculated as total of orifice velocity and terminal velocity of a drop. Deceleration b is calculated as $b = V_{\mathrm{m}}^2/2P$ with the assumption that velocity of the drop reduces from V_{m} at the vena contracta to zero at a location midway between the two neighbouring holes. The calculation of the drop diameter is iterative in this correlation. It involves assuming a drop diameter which is used for calculating its terminal velocity. The assumed values are changed so as to reduce the error between the two sides of Eq. (29).

Kumar and Hartland [27] formulated an empirical correlation by fitting to the experimental data of a large number of investigators.

$$d_{32} = 1.35\sigma^{0.5}\Delta\rho^{-0.5}g^{-0.375}\alpha^{0.4}h^{0.18}\mu_{\mathrm{d}}^{0.14}\sigma_*^{-0.255}\rho_*^{0.055} \times \left[0.23 + \exp\left(-29.66\frac{Af^2}{g\alpha}\right)\right] \quad (30)$$

Here, σ_* (0.072 N/m) and ρ_* (1000 kg/m^3) are the surface tension and density of pure water. This correlation neglects the effect of hole diameter as well as mass transfer on drop size. Its applicability is limited to water as the continuous phase.

Pietzsch and Blass [31] proposed a model to calculate the maximum stable drop size in pulsed column based on energy balance in the column. According to their model, a stable drop will be formed when there is equilibrium between the kinetic energy of the drops in the orifice region and its surface energy. The surface energy of a drop decides energy content of it. The maximum kinetic energy of the drop as it flows through the sieve tray is

$$E_{\mathrm{k\,max}} = \frac{1}{2}V_1^2\rho_{\mathrm{d}}\frac{\pi}{6}d_{\mathrm{p}}^3 \quad (31)$$

The minimum kinetic energy in the flow region outside the sieve trays is

$$E_{\mathrm{k\,min}} = \frac{1}{2}V_2^2\rho_{\mathrm{d}}\frac{\pi}{6}d_{\mathrm{p}}^3 \quad (32)$$

The excess kinetic energy, i.e. the difference between the maximum and minimum kinetic energy must be transferred to the surroundings via the drop surface:

$$\Delta E_{\mathrm{k}} = \frac{1}{12}\rho_{\mathrm{d}}\pi d_{\mathrm{p}}^3(V_1^2 - V_2^2) \quad (33)$$

The cohesion energy of the drops can be calculated from the difference between the surface energy and the curvature pressure:

$$E_\sigma = d_{\mathrm{p}}^2\pi\sigma - \frac{2}{3}d_{\mathrm{p}}^2\pi\sigma = \frac{1}{3}d_{\mathrm{p}}^2\pi\sigma \quad (34)$$

As long as the cohesion energy exceeds the kinetic energy: the drop does not break up. The maximum stable drop diameter can be obtained as

$$d_{\mathrm{p}} = \frac{4\sigma}{\rho_{\mathrm{d}}(V_1^2 - V_2^2)} \quad (35)$$

Thus, the drop size depends on the physical properties and the difference between the squares of velocities. A comparison of the exponents on σ for different correlation in Table 7 shows that the approach of reference [31] shows excessively large dependence of drop size on σ.

Kleczek et al. [32] measured drop size in a 0.067 m diameter column for water and water–glycerine as the continuous phase and various organics as dispersed phase and solutes. They proposed two different correlations depending upon the direction of mass transfer. Drop size calculation using their correlation require a priori knowledge of drop size in absence of solute and dσ/dc, i.e. change in interfacial tension with respect to solute concentration.

$$d_{32} = d_{32}^0 + \frac{2.62\times10^{-8}\,B}{\mu_{\mathrm{c}}^{1.45}}\left(\frac{D_{\mathrm{d}}}{D_{\mathrm{c}}}\right)^{2.0} \quad \text{for d} \rightarrow \text{c} \quad (36)$$

$$d_{32} = d_{32}^0 + \frac{93\,B}{\mu_{\mathrm{c}}^{-1.55}}\left(\frac{D_{\mathrm{d}}}{D_{\mathrm{c}}}\right)^{-1.8} \quad \text{for c} \rightarrow \text{d} \quad (37)$$

where B is given by

For $|I_{\mathrm{M}}| \geq 0.3$; $B = 1$,

For $|I_{\mathrm{M}}| < 0.3$ $\quad B = \dfrac{|I_{\mathrm{M}}|}{0.115 + 0.574|I_{\mathrm{M}}|}$ and

Marangoni index $(I_{\mathrm{M}}) = \dfrac{\Delta c}{\sigma}\dfrac{\mathrm{d}\sigma}{\mathrm{d}c}$

Lorenz et al. [18], measured drop size distribution in columns of 0.080 m diameter. They reported decrease in d_{32} in the direction of flow of the dispersed phase for first two to three sieve plates. This was because of breakage of large drops of the dispersed phase while passing through plate holes. Beyond this the drop diameter was found to vary little along the column height. They observed that drop size in pulsed column is not affected by column diameter. This is because the energy input in pulsed column is uniformly distributed over the cross section of the column.

Kumar and Hartland [33] proposed an empirical formula which takes into account the effect of mass transfer and its direction.

$$d_{32} = C\sigma^{0.5}\Delta\rho^{-0.5}g^{-0.5}\alpha^{0.74}h^{0.1}h_*^{-0.1}\exp\left(-3.00\frac{Af\Delta\rho^{1/4}}{g^{1/4}\sigma^{1/4}}\right) + \exp\left(-28.56\frac{Af\Delta\rho^{1/4}}{g^{1/4}\sigma^{1/4}}\right) \quad (38)$$

Table 7
Variation in the coefficients on different parameters in correlations of drop size in pulsed sieve plate column

Reference	Af	d	α	h	ρ_{c}	ρ_{d}	μ_{c}	μ_{d}	σ	g
Misek [23]	−1.2	0.4	0.3		−0.6				0.6	
Kagan et al. [15]	−0.3				−0.6		0.1		0.5	−0.4
Miyauchi and Oya [9]	−1.2			0.4						
Pilhofer [25]	−1.2			0.4	−0.6					
Boyadzhiev and Spassov [10]	−1.2	0.4	0.8		−0.6				0.6	
Kumar and Hartland [27]			0.4	0.18				0.14	0.56	0.4
Pietzsch and Blass [31]						−1.0			1.0	
Srinivasulu et al. [35]	−0.8	0.26	0.48	0.34	−0.4				0.4	

Here, $h_* = 0.05$ m, $C = 1.51$ for no mass transfer, $C = 1.36$ for c → d and $C = 2.01$ for d → c.

Kumar and Hartland [34] proposed a more comprehensive empirical formula by taking into account large set of experimental data.

$$d_{32} = \frac{C\alpha^{0.32}h^{1.0}}{0.645\sigma^{-0.5}\Delta\rho^{0.5}g^{0.5}h^{1.0} + 2.38\varepsilon^{0.35}\sigma^{-0.66}\Delta\rho^{0.66}g^{0.14}h^{1.15}} \tag{39}$$

where $C = 1$ for no mass transfer, $C = 0.92$ for c → d and $C = 1.67$ for d → c.

Both the above correlations [33,34] do not consider any dependence of drop size on hole diameter. However, drop size is affected by hole diameter as observed from the works of references [10,22,33] in Table 7. The absence of the effect of hole size on drop diameter in the above two correlations could be due to little variation in it in the data used for formulating them.

Srinivasulu et al. [35] measured drop size in 0.043 m diameter column for water–kerosene system. They have reported that at low and medium pulse velocities covering mixer-settler and dispersion regimes multimodal distribution was obtained which changes to unimodal with increase in pulse velocity which results in greater drop breakage on account of increased turbulence. They attributed the drop breakup in the liquid pulsed column to the turbulence due to pulsing, to the breakup while passing through the perforated plates, and to the disintegration on collision with the horizontal plates and with the wall. They observed that the dispersed phase velocity and the continuous phase velocity do not influence the drop size distribution, while drop size increases with increase in hole diameter, fractional free area and plate spacing. They proposed an empirical correlation for calculating drop size in pulsed column.

$$d_{32} = C\sigma^{0.4}\rho_c^{-0.4}(Af)^{-0.8}\alpha^{-0.48}d^{0.26}h^{0.34} \tag{40}$$

The above correlation shows negative exponent on fractional free area (α). However, when their experimental data points were compared with those obtained from their correlation, it was concluded that it should be 0.48 instead of −0.48. This is also in accordance with the general observation as with increase in fractional free area drop coalescence increases due to higher proximity of drops.

Luo et al. [36] studied the drop size variation in a coalescence-dispersion pulsed column with arrangement of one coalescence plate after every four plates. Their system of study was water–TBP–30% kerosene with mass transfer from d → c. They observed that drop size decreases with pulsation intensity as well as along the column height.

2.3.1. Recommended correlation for scale-up

To summarize, an increase in the pulse velocity increases the shear forces on the drops resulting in smaller size of drops. Hole diameter is an important parameter when the column operate in mixer-settler regime. However, its role on drop size decreases as they become smaller than the hole size. An increase in the fractional free area or plate spacing reduces the shear forces on drops resulting in an increase in drop size. Effect of phase throughput on drop size is difficult to comprehend, as there are few studies on this aspect. However, Kagan et al. [15] have shown that varying the phase ratio from 0.1 to 0.5 had no influence on the droplet size.

Experimental data of Jones [22], Khemongkorn et al. [29], Lorenz et al. [18], Srinivasulu et al. [35], Luo et al. [36] and Mohanty and Vogelpohl [37] were used for comparing the different correlations for predicting the drop size in pulsed sieve plate columns. A total of 104 points were obtained from the work of above investigators. Table 6 shows the variation in the experimental data with respect to the geometrical and operating parameters and physical properties.

Correlations were tested for the data satisfying the condition given by respective investigators, e.g. the correlation of Boyadzhiev and Spassov [10] is applicable only for the $\rho_c(2Af)^3/2\alpha^2 > 0.48\ \text{kg/s}^2$. Only 16 points were found to satisfy the above criteria. The force balance model of Pietzsch and Pilhofer [30] uses the correlation of Hu and Kintner [38] to calculate the terminal velocity of a single drop in an infinite medium, V_∞ as a function of drop diameter. For the data sources used, it was observed that the correlation of Hu and Kintner [38] gave negative values of V_∞ when experimentally observed drop diameters were used, so it was not possible to use the method of Pietzsch and Pilhofer [30]. Similar observation has been reported by reference [34]. For understanding the effect of different geometrical and operating parameters the exponents of various parameters reported in the correlations are compared in Table 7.

The effect of continuous phase velocity (V_c) present in the correlation of Misek [23] was neglected for calculating the exponent on Af. However, the effect of V_c was included when drop size was calculated using his equation. Due to complex groups it was not possible to separate the effect of individual parameter in some of the correlations listed in Table 8.

Fig. 5 shows the predictive ability of the four major correlations [23,33–35] formulated over a large data set as compared to others. The correlations due to Misek [23] and Kumar and Hartland [33,34] were observed to overpredict the drop size as shown in Fig. 5a–c. Fig. 5d shows the predictive ability of the correlation given by Srinivasulu et al. [34]. This correlation gives satisfactory prediction of the drop size. The overpredicting tendency of the correlation [23,33,34] can be linked to a larger dependence of drop size on the interfacial tension. As shown in Table 8, for the correlation [23,33,34], $d_{32}\alpha\sigma^{0.6}$ whereas for correlations of Srinivasulu et al. [35] $d_{32}\alpha\sigma^{0.4}$. The correlation of Misek [23] does not take into account the effect of plate spacing on drop size. Also the exponent on plate spacing in the correlations due to Kumar and Hartland [33,34] is around 0.15 which is very low as compared to 0.34 in the correlation of Srinivasulu et al. [35]. Thus, the correlations of Kumar and Hartland [33,34] underestimate the effect of plate spacing on drop size. Misek [23] correlation shows $d_{32}\alpha d^{0.4}$ as compared to $d_{32}\alpha d^{0.26}$ for reference [35]. Whereas none of the correlations of Kumar and Hartland [27,33,34] considers the effect of hole diameter on drop size. Thus, it can be concluded that the predictive ability of the correlation of Srinivasulu et al. [35] is much better than others. Hence, it can be used for prediction of drop size in pulsed sieve plate columns.

Table 8
Correlations for predicting drop size in pulsed sieve plate columns

Reference	Correlations	Remark
Misek [23]	$\frac{d_{32}}{d} = 0.439\left[\frac{\sigma\alpha^{0.5}}{d(\pi Af + V_c)^2\rho_c}\right]^{0.6}$	Shows drop size independent of V_d
Kagan et al. [15]	$d_{32} = 0.92\frac{(Af)^{-0.3}\sigma^{0.5}\mu_c^{0.1}}{\rho_c^{0.6}g^{0.4}}$	Useful for low level of turbulence
Miyauchi and Oya [9]	$d_{32} = 2.03 \times 10^{-5}\left(\frac{Af}{h^{1/3}}\right)^{-1.2}$ for $\frac{Af}{h^{1/3}} > 5.57 \times 10^{-2}\ \mathrm{m^{2/3}/s}$	Not applicable for $Af/h^{1/3} < 5.57 \times 10^{-2}\ \mathrm{m^{2/3}/s}$
Angelino et al. [24]	$d_{32} = C_3\left(\frac{\sigma}{\rho_c}\right)^{0.6}(Af)^{-1.2}$	C_3 depends on column geometry
Pilhofer [25]	$d_{32} = 0.18\left(\frac{\sigma}{\rho_c}\right)^{0.6}(\varepsilon_m)^{-0.4}$	Not applicable for low pulsation levels
Khemongkorn et al. [29]	$d_{32} = 2.59 \times 10^{-5}A^{-1.0}f^{-1.24}N^{-0.21}Vd^{-0.083}$ $d_{32} = 4.90 \times 10^{-5}A^{-1.0}f^{-1.24}N^{-0.21}Vd^{-0.0002}$	c → d d → c
Ugarcic [26]	$d_{32} = C_4\left(\frac{\sigma}{\rho_c}\right)^{0.6}\varepsilon_m^{-0.4}$ $C_4 = 0.132Af$ for $0 < Af \leq 31$ mm/s $C_4 = 0.405$ for 31 mm/s $< Af \leq 59$ mm/s $C_4 = 1.41–0.17Af$ for $59 < Af \leq 80.6$ mm/s	$C_4 = 0.132Af$
Boyadzhiev and Spassov [10]	$d_{32} = 0.57\left(\frac{\sigma}{\rho_c}\right)^{0.6}\frac{\alpha^{0.8}d^{0.4}}{(2Af)^{1.2}}$ for $\frac{\rho_c(2Af)^3}{(2\alpha^2)} > 0.48\ \mathrm{kg/s^2}$	Applicable only in emulsion regime
Logsdail and Slater [21]	$V_0 = V_\infty = \frac{d^2 g\Delta\rho}{18\mu_c(1 + 0.15\,Re_\infty^{0.687})}\quad Re_\infty = \frac{\mathrm{d}V_\infty\rho_c}{\mu_c}$	V_0 is calculated using equation of Thornton [13].
Kumar and Hartland [27]	$\frac{d_{32}}{\sqrt{\sigma/\Delta\rho g}} =$ $1.35\alpha^{0.4}\left(\frac{h}{\sqrt{\sigma_*/\rho_* g}}\right)^{0.18}\left(\frac{\mu_d g^{1/4}}{\rho_*^{1/4}\sigma_*^{3/4}}\right)^{0.14}\left(\frac{\sigma}{\sigma_*}\right)^{0.06}\left[0.23 + \exp\left(-29.66\frac{Af^2}{g\alpha}\right)\right]$	No effect of mass transfer on drop size. Can be used in dispersion and emulsion regime $\rho_* = 998\ \mathrm{kg/m^3}$, $\sigma_* = 0.072$ N/m
Kumar and Hartland [33]	$\frac{d_{32}}{\sqrt{\sigma/\Delta\rho g}} = C\alpha^{0.74}\left(\frac{h}{h^*}\right)^{0.10}\exp\left(-3.00\frac{Af\Delta\rho^{1/4}}{g^{1/4}\sigma^{1/4}}\right) + \exp\left(-28.56\frac{Af\Delta\rho^{1/4}}{g^{1/4}\sigma^{1/4}}\right)$	$C = 1.51$ for no mass transfer. $C = 1.36$ c → d, $C = 2.01$ d → c, $h^* = 0.05$ m
Kumar and Hartland [34]	$\frac{d_{32}}{h} = \frac{C\alpha^{0.32}}{1/1.55(\sigma/\Delta\rho g h^2)^{1/2} + 1/0.42[(\varepsilon_m/g)(\Delta\rho/g\sigma)^{1/4}]^{-0.35}[h(\Delta\rho g/\sigma)^{1/2}]^{-1.15}}$	$C = 1$ for no mass transfer, $C = 0.92$ c → d, $C = 1.67$ d → c
Shreenivasulu et al. [35]	$d_{32} = C\left(\frac{\sigma}{\rho_c}\right)^{0.4}(Af)^{-0.8}\alpha^{0.48}d^{0.26}h^{0.34}$	$C = 0.08$ for no mass transfer, $C = 0.1$ d → c

2.4. *Hold-up of the dispersed phase*

Cohen and Beyer [39] measured hold-up in a column of 0.0254 m diameter for water–boric-acid–*iso*-amyl alcohol. They observed that hold-up increased with an increase in pulse frequency, amplitude and flow rate of either phase. However, the effect of continuous phase velocity on hold-up is small as compared to other parameters.

Logsdail and Thornton [40] conducted experiments with different liquid–liquid systems, column diameter (0.15, 0.23, 0.3 m) and operating conditions and observed that hold-up was independent of the column diameter. They used the slip velocity approach Eq. (4) to put forth a correlation which involves the concept of characteristic velocity (V_0). V_0 can be obtained by using Eq. (5).

Sehmel and Babb [5] measured hold-up in a column of 0.0508 m diameter for three liquid–liquid systems. They studied the effect of frequency and amplitude separately and observed that the minimum hold-up increased slightly with amplitude due to formation of small drops. Hold-up was high at low frequencies (mixer-settler regime) and decreased with increasing frequency until a minimum is reached (dispersion regime). They also showed that hold-up is practically independent of the continuous phase superficial velocity in their operating range. A separate influence of the dispersed phase velocity was not investigated.

Miyauchi and Oya [9] measured hold-up in a column of 0.054 m diameter for water–MIBK system for plate spacing in the range of 0.030–0.070 m. With increase in plate spacing, hold-up was observed to decrease due to reduction in shear forces acting on drops. Based on hold-up data in absence and presence of solute transfer from their own measurements together with those from six published sources they proposed correlations as follows

$$\phi = 4.93 \times 10^2\psi^{0.84}V_d^{2/3}\ \text{for}\ \psi < 0.0031\ \mathrm{m^{11/12}\,s^{-1}},$$

$$\phi = 3.42 \times 10^6\psi^{0.24}V_d^{2/3}\ \text{for}\ \psi > 0.0031\ \mathrm{m^{11/12}\,s^{-1}} \qquad (41)$$

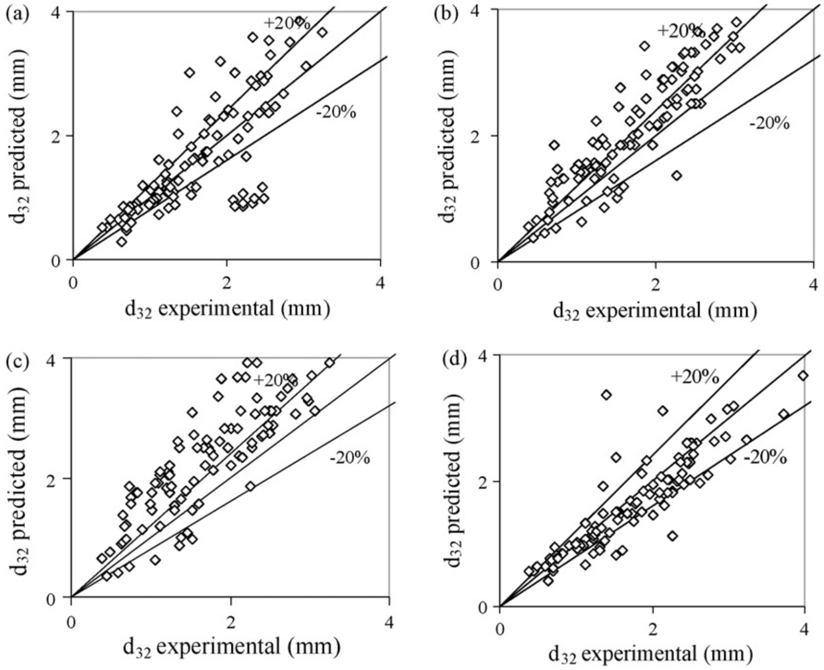

Fig. 5. Testing the predictive ability of different correlations for drop size in pulsed sieve plate columns: (a) Misek et al. [23], (b) Kumar and Hartland [33], (c) Kumar and Hartland [34], and (d) Srinivasulu et al. [35]

$$\psi = \frac{Af}{(\beta h)^{1/3}}\left(\frac{\mu_d^2}{\sigma\Delta\rho}\right)^{1/4} \quad \text{and} \quad \beta = \frac{\alpha^2}{(1-\alpha)(1-\alpha^2)} \tag{42}$$

The above equations show a non-linear relationship between hold-up (ϕ) and superficial velocity dispersed phase (V_d). Furthermore, the effect of V_c on ϕ was found to be insignificant.

Mishra and Dutt [41] measured hold-up of organic phase for toluene–water system in a column of 0.0762 m diameter as a function of pulse velocity, phase flow rates, hole diameter, percentage free area and plate spacing. In the range of variables studied, it was concluded that the hold-up increased with increase of pulse velocity and dispersed phase flow rate, whereas it decreased with increase of hole diameter, percent free area and plate spacing. They proposed the following correlation for toluene–water system. Since the constant C depends on physical properties and operating conditions, experimental investigations are required of its value for other liquid–liquid systems.

$$\phi = CA^{0.81}f^{0.81}h^{-0.27}V_d^{0.89}d^{-0.924}\beta^{-0.27} \tag{43}$$

Bell and Babb [42] measured hold-up for two systems in a column of 0.0508 m diameter. They studied the axial distribution of hold-up for water–hexane system. Hold-up was found to be practically independent of continuous phase velocity, but it increased rapidly with dispersed phase velocity. They formulated a correlation which was able to predict hold-up data for all regimes under the range of studied operating conditions.

$$\phi = 4.19 \times 10^2 V_d(C_1 + (69.75 + 1.59 \times 10^4 V_c)(Af - C_2)^2 \tag{44}$$

where $C_1 = 0.03$ and $C_2 = 0.027$ m/s for n-hexane–water, and $C_1 = 0.05$ and $C_2 = 0.016$ m/s for MIBK–water. Experimental investigation is required for calculating the values of C_1 and C_2 for other liquid–liquid systems.

Khemongkorn et al. [29] measured hold-up for water–iodine–CCl$_4$ system in a 0.050 m diameter column. Hold-up increased with both amplitude and frequency. They found that the group $Af^{1.24}$ correlated their hold-up data more satisfactorily than Af product. Except these authors all other investigators find that the pulse velocity (Af) adequately represents the effect of amplitude and frequency on hold-up over the entire range of agitation. Hold-up for both the directions of mass transfer were reported to be of the same order when A or f is small. They correlated their data on hold-up resulting in different correlations depending on operational regime and direction of mass transfer.

- For mass transfer from c → d

$$\phi = 1.01 \times 10^3 A^{1.26} f^{1.56} V_d Af^{1.24} < 0.055\,\text{m/s}^{1.24} \quad \text{transition regime} \tag{45}$$

$$\phi = 7.69 \times 10^3 A^{1.26} f^{3.6} V_d^{2/3} Af^{1.24} \geq 0.055\,\text{m/s}^{1.24} \quad \text{emulsion regime} \tag{46}$$

- From mass transfer from d → c

$$\phi = 1.53 \times 10^3 A^{1.26} f^{1.71} V_d Af^{1.24} < 0.052\,\text{m/s}^{1.24} \quad \text{transition regime} \tag{47}$$

$$\phi = 3.94 \times 10^4 A^{1.26} f^{4.29} V_d^{2/3} Af^{1.24} \geq 0.052\,\text{m/s}^{1.24} \quad \text{emulsion regime} \tag{48}$$

Kumar and Hartland [11] collected the experimental data of a lot of investigators and correlated to give three empirical correlations depending on the regime of operation. The correlations do not include the effect of mass transfer on hold-up.

$$\phi = 3.91 \times 10^{-3} A^{-0.52} f^{-0.76} \rho_{\mathrm{c}}^{1.15} g^{-0.1} \Delta\rho^{-0.81} \mu_{\mathrm{d}}^{-0.52} \sigma^{0.18} \times V_{\mathrm{d}}^{1.12} \left(1 + \frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.14} \quad \text{mixer-settler} \tag{49}$$

$$\phi = 6.91 A^{0.93} f^{0.93} \rho_{\mathrm{c}}^{1.18} g^{-0.7} \Delta\rho^{-0.79} \mu_{\mathrm{d}}^{-0.04} \sigma^{-0.35} V_{\mathrm{d}}^{1.2} \times h^{-0.31} \beta^{-0.31} \left(1 + \frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.14} \quad \text{transition} \tag{50}$$

$$\phi = 3.73 \times 10^{-3} A^{1.24} f^{1.24} \rho_{\mathrm{c}}^{3.11} g^{-0.91} \Delta\rho^{-2.20} \mu_{\mathrm{d}}^{-1.16} \sigma^{0.25} V_{\mathrm{d}}^{1.24} \times \left(1 + \frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.45} \quad \text{emulsion} \tag{51}$$

Tung and Leucke [12] collected the experimental data in emulsion regime. They classified the data in emulsion based to satisfy the criteria $\rho_{\mathrm{c}}(Af)^3/(2\alpha^2) > 0.06\,\mathrm{kg/s^2}$. They put forth the following correlation for hold-up in this regime.

$$\phi = C A^{1.90} f^{1.90} \mu_{\mathrm{d}}^{0.72} \sigma^{-0.36} \Delta\rho^{-0.36} \beta^{-0.63} h^{-0.63} V_{\mathrm{d}}^{1.1} \tag{52}$$

where $C = 3.98 \times 10^5$ for no solute transfer and $C = 2.52 \times 10^5$ for solute transfer in either direction.

Hussain et al. [43] formulated the following correlation for characteristic velocity (V_0) by their study over different systems and operating conditions. This correlation is applicable only when drop size is smaller than hole diameter. V_0 obtained from this equation can be used for calculating hold-up using the equation of V_{slip}.

$$\frac{V_0}{V_\infty} = 1 - \frac{d_{32}}{[1 + 100(0.275 + 0.365\alpha)h]d} - \left[\frac{Af}{0.01 + 0.78h} - \frac{100(Af)^2}{0.01 + 2.6h}\right] \quad \text{for } d_{32} < d \tag{53}$$

V_∞ is calculated using equation of Grace et al. [44], which requires d_{32}. Hence, it requires a priori knowledge of drop size.

Pietzsch and Blass [31] measured hold-up in a column of 0.072 m diameter for water as continuous phase and a mixture of TBP and *n*-alkane as dispersed phase. They measured hold-up under different operating conditions and summarize that in the mixer-settler regime drops accumulate below the plate and hold-up is high. Hold-up reaches a minimum value when transition from mixer-settler to dispersion regime occurs. Hold-up increases with an increase in pulse velocity in this regime. As the upper flooding limit is approached, hold-up increases strongly and smallest drop fractions are washed out by the continuous phase.

Kumar and Hartland [7] presented a correlation by fitting to the experimental data of a large number of investigators. They have also listed different correlations for V_0 reported by different investigators. They have shown that the correlation can be applied for all regimes of operation of the pulsed column.

$$\phi = K_1 \exp[K_2 |Af - (Af)_{\mathrm{m}}|] V_{\mathrm{d}}^{0.86} (V_{\mathrm{c}} + V_{\mathrm{d}})^{0.28} \Delta\rho^{-0.30} \rho_{\mathrm{d}}^{-0.93} \times \mu_{\mathrm{d}}^{0.77} \alpha^{-0.56} h^{-0.56} \tag{54}$$

$$(Af)_{\mathrm{m}} = 9.69 \times 10^{-3} \left(\frac{\sigma \Delta\rho^{1/4} \alpha}{\mu_{\mathrm{d}}^{3/4}}\right)^{0.33}$$

where $K_1 = 2.10 \times 10^6$ for no solute transfer, $K_1 = 2.14 \times 10^6$ for c → d and $K_1 = 1.10 \times 10^6$ for d → c, and $K_2 = 44.53$ for no solute transfer, $K_2 = 44.53$ for c → d and $K_2 = 50.56$ for d → c.

Kumar and Hartland [33] developed empirical correlations for slip velocity in terms of the physical properties of liquid–liquid systems, column geometries and operating conditions. This obviates knowledge of hold-up beforehand for calculating slip velocity.

$$V_{\mathrm{slip}} = K_1 \exp[K_2 |Af - (Af)_{\mathrm{m}}|] \Delta\rho^{0.29} \rho_{\mathrm{d}}^{0.67} \mu_{\mathrm{d}}^{-0.66} \alpha^{0.44} h^{0.43} \tag{55}$$

where $K_1 = 6.14 \times 10^{-6}$ for no solute transfer, $K_1 = 5.04 \times 10^{-6}$ c → d and $K_1 = 6.43 \times 10^{-6}$ for d → c, and $K_2 = -36.91$ for no solute transfer, $K_2 = -30.79$ for c → d and $K_2 = -31.81$ for d → c.

Lorenz et al. [18] measured hold-up for three systems without mass transfer effects in a column of 0.080 m diameter. Under any operating conditions, the local hold-up was observed to increase continuously in the flow direction of the dispersed phase on account of decreasing drop size and the associated decline in the relative velocity of the drops. The throughput of the dispersed phase was found to exert a far stronger influence on the hold-up than that of the continuous phase. Thus, at a constant total throughput, the hold-up increased with increasing phase ratio, $V_{\mathrm{d}}/V_{\mathrm{c}}$.

Mohanty and Vogelpohl [37] measured hold-up for water–butyl acetate system in a column of 0.080 m diameter. They observed that hold-up increased with an increase in the pulse velocity. They also observed that hold-up in absence of coalescence is more than in presence of it. This is because larger drops formed due to coalescence have high rising velocity resulting in less hold-up.

Venkatnarasaiah and Verma [45] measured hold-up in water–kerosene system with *n*-butyric acid and benzoic acid as solute. The mass transfer direction was d → c. Dispersed phase hold-up decreased with increase in pulse velocity in the mixer-settler regime, attained a minimum at the transitional pulse velocity ($Af = 0.015$–0.02 m/s) beyond which it increased with increase in pulse velocity The data showed a profound influence of the perforation diameter and free area of the plates and plate spacing on the dispersed phase hold-up. It was observed that an increase in the hole diameter, free area and plate spacing decreases the dispersed phase hold-up considerably. They also observed very small effect of V_{c} on hold-up. They gave a correlation to calculate hold-up in pulsed columns.

$$\phi = K_1 \exp[K_2|Af-(Af)_{\mathrm{m}}|]V_{\mathrm{d}}^{1.02}V_{\mathrm{c}}^{0.02}\Delta\rho^{-0.23}\mu_{\mathrm{d}}^{0.52}d^{-0.3} \times \alpha^{-0.4}h^{-0.4} \qquad (56)$$

where $K_1 = 116.5$ for no solute transfer, $K_1 = 84.6$ for c → d and $K_1 = 92.0$ for d → c, and $K_2 = 39.35$ for no solute transfer, $K_2 = 42.56$ for c → d and $K_2 = 42.56$ for d → c.

They also formulated a correlation for direct evaluation of V_{slip} as follows.

$$V_{\mathrm{slip}} = K_1 \exp[K_2|Af-(Af)_{\mathrm{m}}|]\Delta\rho^{0.22}\mu_{\mathrm{d}}^{-0.38}\alpha^{0.32}h^{0.31}d^{0.22} \qquad (57)$$

where $K_1 = 1.35 \times 10^{-2}$ for no solute transfer, $K_1 = 1.65 \times 10^{-2}$ c → d and $K_1 = 1.55 \times 10^{-2}$ for d → c, and $K_2 = -33.3$ for no solute transfer, $K_2 = -29.6$ for c → d and $K_2 = -29.6$ for d → c.

2.4.1. Recommended correlation for scale-up

To summarize, hold-up in the mixer-settler regime decreases with increase in pulse velocity until a minimum is reached. This minimum corresponds to the transition from mixer-settler to dispersion regime. Beyond this minimum, hold-up increases with pulse velocity. Continuous increase in pulse velocity finally leads to flooding. Hole diameter is an important parameter when the column operate in mixer-settler regime. However, its role on hold-up decreases as drops become smaller than the hole size. An increase in the fractional free area or plate spacing reduces the shear forces on drops resulting in an increase in drop size which leads to reduction in hold-up. Hold-up is a strong function of the dispersed phase velocity (V_{d}) and an increase in V_{d} increases hold-up. However, effect of continuous phase velocities (V_{c}) on hold-up has been found be negligible.

A total of 247 experimental data points for hold-up in pulsed sieve plate extraction column were obtained from the work of Bell and Babb [42], Lorenz et al. [18], Mohanty and Vogelpohl [37] and Venkatnarasaiah and Verma [45]. Table 6 shows the variation in the experimental data with respect to the geometrical and operating parameters and physical properties.

A large number of correlations for hold-up are reported in the literature. Kumar and Hartland [7] have reviewed them and formulated a new correlation which they claim is more general than previously reported ones. Two major correlations for hold-up are that of Kumar and Hartland [7] and Venkatnarasaiah and Verma [45]. The predictive ability of these two correlations is shown in Fig. 6a and c. It can be observed that both the correlations overpredict the hold-up value. The nature of the spread of the values for the two correlations is same. This is because of their similar form as shown in Table 9. Thus, none of them are good enough to predict hold-up. Kumar and Hartland [32] and Venkatnarasaiah and Verma [45] gave correlations for directly calculating slip velocity $V_{\mathrm{slip.}}$ These were used for calculating V_{slip} and hold-up was calculated from it using Eq. (4). The results shown in Fig. 6b and d indicate that the V_{slip} correlation of Venkatnarasaiah and Verma [45] has good ability to predict hold-up as compared to Kumar and Hartland [32]. This could be because the

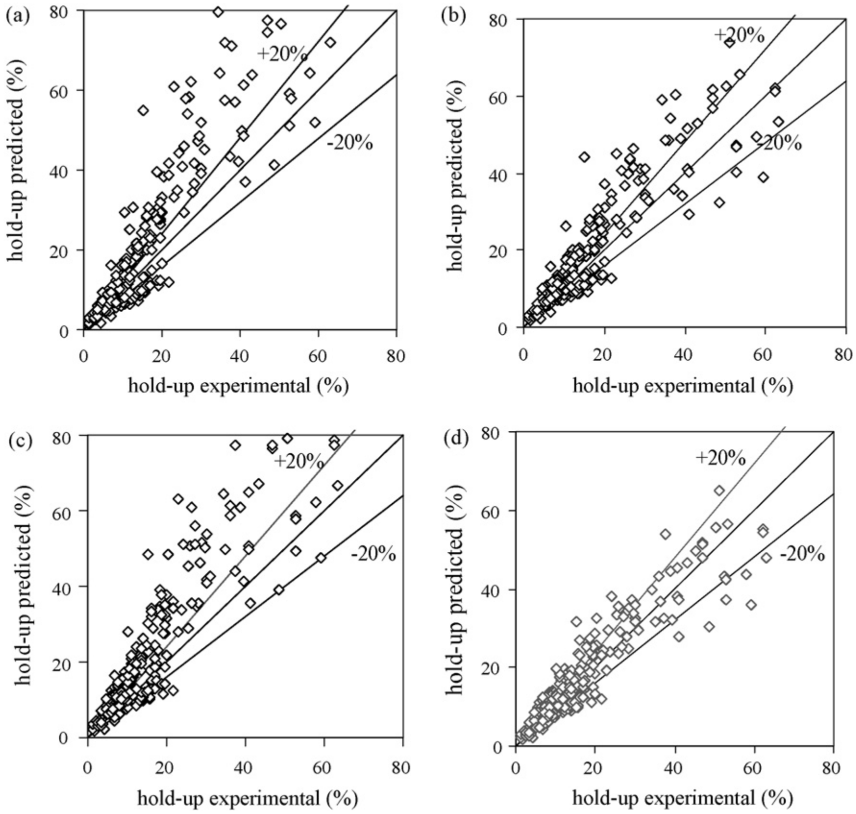

Fig. 6. Testing the predictive ability of different correlations for hold-up in pulsed sieve plate columns: (a) Kumar and Hartland [7], (b) Kumar and Hartland [33], (c) Venkatnarsaiah and Verma [45], and (d) Venkatnarsaiah and Verma [45]

Table 9
Correlation for predicting hold-up in pulsed sieve plate columns

Investigators	Correlations	Remark		
Miyauchi and Oya [9]	$\phi = 4.93 \times 10^{2}\psi^{0.84}V_{\mathrm{d}}^{2/3}$ for $\psi < 0.0031\,\mathrm{m}^{11/12}\,\mathrm{s}^{-1}$, $\phi = 3.42 \times 10^{6}\psi^{0.24}V_{\mathrm{d}}^{2/3}$ for $\psi > 0.0031\,\mathrm{m}^{11/12}\,\mathrm{s}^{-1}$, $\beta = \dfrac{\alpha^2}{(1-\alpha)(1-\alpha^2)}$	$\psi = \dfrac{Af}{(\beta h)^{1/3}}\left(\dfrac{\mu_{\mathrm{d}}^2}{\sigma\Delta\rho}\right)^{1/4}$		
Kumar and Hartland [11]	$\phi = 3.91 \times 10^{-3}\left(\dfrac{A^2\rho_{\mathrm{c}}g}{\sigma}\right)^{-0.26}\left(\dfrac{f^4\sigma}{\rho_{\mathrm{c}}g^3}\right)^{-0.19}\left(\dfrac{V_{\mathrm{d}}^4\rho_{\mathrm{c}}}{g\sigma}\right)^{0.28}\left(1+\dfrac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.19}\left(\dfrac{\Delta\rho}{\rho_{\mathrm{c}}}\right)^{-0.81}\left(\dfrac{\mu_{\mathrm{d}}^4 g}{\rho_{\mathrm{c}}\sigma^3}\right)^{-0.13}$	Mixer-settler		
	$\phi = 6.91\left[\dfrac{(Af)^3\rho_{\mathrm{c}}^{1/4}}{\beta h\sigma^{1/4}g^{5/4}}\right]^{0.31}\left(\dfrac{V_{\mathrm{d}}^4\rho_{\mathrm{c}}}{g\sigma}\right)^{0.30}\left(1+\dfrac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.14}\left(\dfrac{\Delta\rho}{\rho_{\mathrm{c}}}\right)^{-0.79}\left(\dfrac{\mu_{\mathrm{d}}^4 g}{\rho_{\mathrm{c}}\sigma^3}\right)^{-0.01}$	$\left[\dfrac{(Af)^3\rho_{\mathrm{c}}}{\beta h\Delta\rho^{3/4}\sigma^{1/4}g^{5/4}}\right] < 0.05$ transition		
	$\phi = 3.73 \times 10^{-3}\left[\dfrac{(Af)^4\rho_{\mathrm{c}}}{g\sigma}\right]^{0.31}\left(\dfrac{V_{\mathrm{d}}^4\rho_{\mathrm{c}}}{g\sigma}\right)^{0.31}\left(1+\dfrac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.45}\left(\dfrac{\Delta\rho}{\rho_{\mathrm{c}}}\right)^{-2.20}\left(\dfrac{\mu_{\mathrm{d}}^4 g}{\rho_{\mathrm{c}}\sigma^3}\right)^{-0.29}$	$\left[\dfrac{(Af)^3\rho_{\mathrm{c}}}{\beta h\Delta\rho^{3/4}\sigma^{1/4}g^{5/4}}\right] > 0.05$ emulsion		
Tung and Luecke [12]	$\phi = C\left[\dfrac{Af}{(\beta h)^{1/3}}\right]^{1.90}\left(\dfrac{\mu_{\mathrm{d}}^2}{\sigma\Delta\rho}\right)^{0.36}V_{\mathrm{d}}^{1.1}$ for $\dfrac{\rho_{\mathrm{c}}(Af)^3}{(2\alpha^2)} > 0.06\,\mathrm{kg/s^2}$	$C = 3.98 \times 10^5$ for no solute transfer, $C = 2.52 \times 10^5$ for solute transfer in either direction		
Kumar and Hartland [7]	$\phi = K_1\exp[K_2\lvert Af-(Af)_{\mathrm{m}}\rvert]V_{\mathrm{d}}^{0.86}(V_{\mathrm{c}}+V_{\mathrm{d}})^{0.28}\Delta\rho^{-0.30}\rho_{\mathrm{d}}^{-0.93}\mu_{\mathrm{d}}^{0.77}$, $\alpha^{-0.56}h^{-0.56}$, $(Af)_{\mathrm{m}} = 9.69 \times 10^{-3}\left(\dfrac{\sigma\Delta\rho^{1/4}\alpha}{\mu_{\mathrm{d}}^{3/4}}\right)^{0.33}$		Condition K_1	Condition K_2
		No solute transfer	2.10×10^6	44.53
		c → d transfer	2.14×10^6	44.53
		d → c transfer	1.10×10^6	50.56
Venkatnarasaiah and Verma [45]	$\phi = K_1\exp[K_2\lvert Af-(Af)_{\mathrm{m}}\rvert]V_{\mathrm{d}}^{1.02}V_{\mathrm{c}}^{0.02}\Delta\rho^{-0.23}\mu_{\mathrm{d}}^{0.52}d^{-0.3}\alpha^{-0.4}h^{-0.4}$		Condition K_1	Condition K_2
		No solute transfer	116.5	39.35
		c → d transfer	84.6	42.56
		d → c transfer	92.0	42.56

V_{slip} correlation of Kumar and Hartland [32] does not consider effect of hold diameter on hold-up. Whereas V_{slip} correlation of Venkatnarasaiah and Verma [45] considers $V_{\text{slip}} \alpha d^{0.22}$. The V_{slip} correlation of Kumar and Hartland [32] also overestimate the effect of physical properties of the dispersed phase on V_{slip}. Hence, the V_{slip} correlation of Venkatnarasaiah and Verma [45] is recommended for calculating hold-up in pulsed sieve plate extraction columns.

2.5. Mass transfer in pulsed columns

Mass transfer in pulsed column has been studied by many workers ranging from Cohen and Beyer [39] and Sege and Woodfield [3] to He et al. [46]. In most cases mass transfer performance has been reported in terms of overall mass transfer coefficients or $K_{\text{oc}}\underline{a}$ without correction for axial mixing effects. Buratti [47] cited from Nabli et al. [48] have reported that overlooking the effect of axial mixing when designing a column can lead to an overprediction of the mass transfer efficiency between phases by about 30%.

Cohen and Beyer [39] studied the variation of $K_{\text{oc}}\underline{a}$ with phase flow rates, pulse amplitude and frequency, capacity and hold-up using water (continuous)–boric acid–*iso*-amyl alcohol (dispersed). The mass transfer was from dispersed to continuous ($\text{d} \rightarrow \text{c}$) phase. They observed that, $K_{\text{oc}}\underline{a}$ increased with an increase in dispersed phase flow rate, pulse amplitude and frequency. They also found better mass transfer rates for water continuous than organic.

Sege and Woodfield [3] studied solvent extraction of uranyl nitrate in 0.0762 m diameter column. $K_{\text{oc}}\underline{a}$ was observed to increase with increase in pulse velocity. However, for a large pulse velocity appreciable axial mixing occurred and mass transfer effectiveness of the column was observed to decrease. They found no scale-up effect between 0.075 and 0.2 m diameter column on $K_{\text{oc}}\underline{a}$ but reported a four-fold decrease when the column diameter was increased from 0.2 to 0.6 m. The reduction in the mass transfer coefficient can be attributed to increase in the axial mixing in the column. Another factor of this observation could be the limitations of their mechanical pulsing device. Even though they have increased the diameter of the column, the diameter of the pulsing section was kept constant. Such an arrangement will definitely increase the non-uniformity of flow across the column with increase in its diameter.

Chantry et al. [49] studied the effect of pulsation on mass transfer effectiveness of a packed column and a sieve plate column. He concluded that for the packed column a three-fold increase in $K_{\text{oc}}\underline{a}$ is obtained when pulsation is applied to it. For sieve plate columns he reported an increase of as high as 70% in the mass transfer coefficients.

Li and Newton [50] studied the extraction of benzoic acid from dispersed toluene phase using water in a column of 0.0508 m diameter. They suggested that the performance of the sieve plate column can be greatly improved by superimposing pulsations. They found a critical Reynolds number, 1200 based on jet velocity and hole diameter above which the rate of increase of the mass transfer coefficient was much more rapid with increase in Reynolds number. The physical properties (ρ and μ) for calculating N_{Re} were based on the properties of the mixture by taking an average hold-up of 16%. This Reynolds number marked the transition of flow from streamwise to turbulent conditions. The critical N_{Re} was found to decrease with increase in flow rates of the phases. Also the column was observed to flood at a Reynolds number of 1800.

Thornton [13] applied the concept of characteristic velocity (V_0) for the prediction of $K_{\text{oc}}\underline{a}$. For the experimental data obtained for extraction of acetone from toluene using water in a column of 0.0762 m diameter he correlated his data using dimensionless groups.

$$K_{\text{oc}}\underline{a} = V_{\text{c}}\left[b\left(\frac{\mu_{\text{c}}^2}{g\rho_{\text{c}}^2}\right)^{0.33}\left(\frac{\mu_c g}{V_0^3(1-\phi)^3\rho_{\text{c}}}\right)^{2n/3}\left(\frac{\Delta\rho}{\rho_{\text{c}}}\right)^{2(n-1)/3} \times \left(\frac{V_{\text{d}}}{V_{\text{c}}}\right)^{0.5}\left(\frac{V_{\text{c}}^3\rho_{\text{c}}}{g\mu_{\text{c}}\phi^3}\right)^{0.33}\right]^{-1} \quad (58)$$

The above correlation for $K_{\text{oc}}\underline{a}$ is uncorrected for axial mixing. The range of $A \times f$ was 10–50 mm/s. Experimental measurements are needed to determine n and b.

Logsdail and Thornton [40] found a 30% decrease in $K_{\text{oc}}\underline{a}$ on increasing the diameter from 0.15 to 0.3 m because of increase in axial mixing. This decrease in mass transfer efficiency of the column can be linked to increase in radial non-uniformities of the concentration which increases the dispersion coefficient. These are gain due to limitations in the pulsing device as discussed above. They proposed the correlation $K_{\text{oc}}\underline{a} \alpha V_{\text{c}}[\exp(D/2)]^{-1}$, but this would seriously overestimate the effect for diameters greater than 0.3 m.

Smoot et al. [4] based on 285 data points by various investigators gave correlation for $K_{\text{oc}}\underline{a}$ uncorrected for axial mixing. It applies to the emulsion regime and steel sieve plates. It is valid only for mass transfer from the solvent dispersed phase to an aqueous continuous phase and for the case in which the major resistance to mass transfer is in the dispersed phase.

$$K_{\text{oc}}\underline{a} = 0.096 f^{0.43} A^{0.43} d^{0.43} \rho_{\text{d}}^{2.34} \mu_{\text{d}}^{3.27} V_{\text{d}}^{0.64} D^{-0.32} D_{\text{v}}^{0.865} \times h^{-0.68} \sigma^{-0.097} \Delta\rho^{-1.04} \alpha^{0.43} \quad (59)$$

Sobotic and Himmelblau [51] studied the effect of plate wetting characteristics on pulsed column extraction efficiency. They observed that for mass transfer from the continuous ketone phase to dispersed water, an all polyethylene plate arrangement provided the best efficiency while an all stainless steel plate arrangement was found to be less favourable under same operating conditions. A combination of these two arrangements in the column produced efficiencies midway between the all plastic plate arrangement and the all stainless steel plate arrangement. Thus, $K_{\text{oc}}\underline{a}$ is a strong function of material of construction of plate. The above observation could be related to the wetting characteristics of plastic plates by continuous ketone phase. Stainless steel plates were not appeared to be as easily wetted by water as the plastic plates were wetted by the ketone. The plastic plates were wetted by the ketone so well that a layer of ketone adhered to them even under high pulsations and with water as continuous phase. Thus, the phase not wetting the plate only contacted the wetted phase and not the plate itself.

Smoot and Babb [52] studied the mass transfer in a pulsed column using MIBK–acetic acid–water. The organic phase was dispersed and solute transfer was from the dispersed to the continuous phase (d → c). They concluded that $K_{\mathrm{oc}}\underline{a}$ decreased with an increase in the continuous phase velocity (V_{c}), hole diameter (d) and plate spacing (h). However, it increased with an increase in f, A and V_{d}. The $K_{\mathrm{oc}}\underline{a}$ values have been corrected for the axial mixing and a correlation has been proposed. The correlation does not take into account the effect of interfacial tension and percentage free area. Also the operating conditions have not been mentioned clearly by them.

$$K_{\mathrm{oc}}\underline{a} = 504 f^{0.83} A^{0.83} h^{-0.38} d^{-0.22} \rho_{\mathrm{d}}^{0.4} \mu_{\mathrm{d}}^{-0.4} V_{\mathrm{c}}^{0.01} V_{\mathrm{d}}^{0.56} \tag{60}$$

Angelino et al. [24] used pulsed column equipped with stirrer in each plate section. They observed increased efficiency of this arrangement over that standard pulsed column. However, this arrangement becomes complex due to increased maintenance of stirrer blades.

Khemongkorn et al. [29] studied $K_{\mathrm{oc}}\underline{a}$ as a function of Af. It increased and reached a maximum as the product Af increases. They also observed that an increase in the dispersed phase flow rate increases $K_{\mathrm{oc}}\underline{a}$. They also reported that mass transfer direction has very little effect on $K_{\mathrm{oc}}\underline{a}$ for their system.

Berger et al. [6] studied the effect of interfacial tension and plate geometry on separation efficiency of pulsed column in terms of height equivalent to theoretical stage (HETS). They observed a large effect of interfacial tension on separation efficiency. For large interfacial tension, the efficiency was a strong function of plate geometry, however, this dependence lowered as interfacial tension decreased.

Bahmanyar et al. [53] studied a pulsed column of 0.076 m diameter. The experimental results incorporated some corrections for mass transfer while drop formation.

Vatanatham et al. [55] studied the extraction of acetone from toluene using water. They correlated overall mass transfer coefficient as a function of $N_{Re} = DV\rho_{\mathrm{c}}/\mu_{\mathrm{c}}$ in the range of (2–20). It was found to be proportional to N_{Re}.

$$K_{\mathrm{oc}}\underline{a} = 7.2 \times 10^{-7} N_{Re}^{1.04} \tag{61}$$

Li et al. [56] studied the effect of these coalescence-dispersion sieve plates on mass transfer with the arrangement of one coalescence sieve plate installed after every four plates. The material of construction of the coalescence plates is such that the dispersed phase drops wet this plate. Thus, a coalescence layer is obtained below every such plate. This arrangement tries to take the advantage of the mass transfer during drop formation. They obtained an increase of 30% in efficiency of this column as compared to standard arrangement.

Chao-Hong-He [46] studied mass transfer efficiency of a 0.6 m diameter column used for extraction of caprolactum from waste water. They found that $K_{\mathrm{oc}}\underline{a}$ decreased with a decrease in the continuous phase velocity but it increased with an increase in pulse velocity and dispersed phase velocity. They correlated $K_{\mathrm{oc}}\underline{a}$ (Table 10) with operating conditions. However, the constants were found be a strong function of column diameter.

Kumar and Hartland [57] reviewed various correlations for mass transfer from drops. They extended this work to predict the Sherwood numbers for the dispersed and continuous phases.

Tung and Luecke [12] classified the drops based on a transition drop sizes calculated on the basis of energy dissipated in the column. For drop sizes smaller than the transition size, they suggested the use of rigid drop model of Newman [58] with $k_{\mathrm{d}} = 2\pi^2/3(D_{\mathrm{d}}/d_{32})$. For drop sizes larger than transition drop size, a turbulent mass transfer can be assumed from the drops and hence Handlos and Baron [59] equation $k_{\mathrm{d}} = 0.00375V_{\mathrm{slip}}/1 + \mu_{\mathrm{d}}/\mu_{\mathrm{c}}$ may be used.

Reissinger and Marr [60] cited from Haverland and Slater [61] have recommended the use of Handlos and Baron [59] equation for prediction of drop side mass transfer coefficient. They suggested using the Heertjes et al. [62] correlation for continuous phase. Theses equations simply give the highest expected values of the film mass transfer coefficient and may be the result of the effect of pulsation. However, contamination may be present in the industrial systems and lower values may be more realistic.

2.5.1. Recommended correlation for scale-up

The geometrical and operating parameters of the pulsed column affect its mass transfer effectiveness through their effect on drop size and hold-up. As an increase in the pulse velocity in the mixer-settler regime decreases the hold-up, it reduces the interfacial area, $\underline{a} = 6\phi/d_{32}$. This leads to reduction in mass transfer coefficient, thus necessitating increase in column height. Increase in hole diameter, fractional free area or plate spacing also has a similar effect. Increase in hold-up beyond mixer-settler regime increases the interfacial area thus increasing mass transfer efficiency of the column. However, at very high pulse velocities effect of axial mixing becomes dominant. This decreases the mass transfer effectiveness.

A total of 274 experimental data points for overall height of transfer unit ($\mathrm{HTU_{oc}}$) were taken from the work of Cohen and Beyer [39], Sobotic and Himmelblau [51], Smoot and Babb [52], Venkatnarsaiah and Verma [45], Li et al. [56], He et al. [47] and Usman et al. [63]. Table 6 shows the variation in the experimental data with respect to the geometrical and operating parameters and physical properties.

A number of correlations reported in the literature were tested. These can be classified into two categories: (a) those which directly relate height of transfer units (HTUoc) to operating and geometrical conditions and physical properties [4,52] and (b) those which involve the knowledge of hold-up a priori for calculating HTUoc [13,54]. As with previous parameters, we looked for a generalized correlation in the beginning. Smoot et al. [4] proposed a correlation in terms of dimensionless groups using the experimental data of different investigators. So initially it was tried to increase the generality of their correlation. For this the multiple regression technique was applied to the dimensionless groups of Smoot et al. [4] to calculate the exponents. A negative exponent on the (D/h) term of the correlation (Table 10) was obtained which indicate that the column efficiency increases with increase in its diameter. This is against the

Table 10
Correlations for predicting mass transfer coefficient in pulsed sieve plate columns

Investigators	Correlations	Remark
Thornton [13]	$K_{\mathrm{oc}}\underline{a} = V_{\mathrm{c}}\left[b\left(\frac{\mu_{\mathrm{c}}^2}{g\rho_{\mathrm{c}}^2}\right)^{0.33}\left(\frac{\mu_{\mathrm{c}}g}{V_0^3(1-\phi)^3\rho_{\mathrm{c}}}\right)^{2n/3}\left(\frac{\Delta\rho}{\rho_{\mathrm{c}}}\right)^{2(n-1)/3}\left(\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}\right)^{0.5}\left(\frac{V_{\mathrm{c}}^3\rho_{\mathrm{c}}}{g\mu_{\mathrm{c}}\phi^3}\right)^{0.33}\right]^{-1}$	$m=0.5$ for c → d, $m=0.25$ d → c, b is system specific and should be found experimentally
Smoot et al. [4]	$K_{\mathrm{oc}}\underline{a} = 0.20\left[\left(\frac{V_0 d\rho_{\mathrm{d}}}{\mu_{\mathrm{d}}}\right)^{-0.434}\left(\frac{\Delta\rho}{\rho_{\mathrm{d}}}\right)^{1.04}\left(\frac{\mu_{\mathrm{d}}}{\rho_{\mathrm{d}}D_{\mathrm{v}}}\right)^{0.865}\left(\frac{\sigma}{\mu_{\mathrm{c}}V_{\mathrm{c}}}\right)^{0.096}\left(\frac{V_{\mathrm{d}}}{V_{\mathrm{c}}}\right)^{-0.636}\left(\frac{D}{h}\right)^{0.317}\left(\frac{\mu_{\mathrm{c}}}{\mu_{\mathrm{d}}}\right)^{4.57}\right]^{-1}\left(\frac{V_{\mathrm{c}}}{h}\right)$	Applicable for d → c and water continuous system with stainless steel plates: $V_0 = \frac{Af}{\alpha}$
Smoot and Babb [52]	$K_{\mathrm{oc}}\underline{a} = 504V_{\mathrm{c}}\left[h\left(fA_{\mathrm{p}}\frac{d_0\rho_{\mathrm{d}}}{\mu_{\mathrm{d}}}\right)^{-0.4}\left(\frac{V_{\mathrm{c}}}{fA_{\mathrm{p}}}\right)^{0.43}\left(\frac{V_{\mathrm{c}}}{V_{\mathrm{d}}}\right)^{0.56}\left(\frac{d_0}{h}\right)^{0.62}\right]^{-1}$	Operating conditions are not mentioned properly
Luo et al. [54]	$K_{\mathrm{oc}}\underline{a} = \left[K\frac{1}{\phi(1-\phi)^n}\right]^{-1}\ K=K_1/C\ C=0.52$	$K=K_1/C$, $C=0.52$, $K=25.86$
	$K_1 = \frac{(3\mu_{\mathrm{d}}+2\mu_{\mathrm{c}})}{(3\mu_{\mathrm{d}}+3\mu_{\mathrm{c}})}\left(\frac{4}{225}\frac{\Delta\rho^2g^2}{\rho_{\mathrm{c}}\mu_{\mathrm{c}}}\right)^{-1/3}\frac{m(1+\mu_{\mathrm{d}}/\mu_{\mathrm{c}})}{6\times 0.00375}$	$n = 0.67 + 0.028\left(\frac{\mu_{\mathrm{c}}}{\mu_{\mathrm{d}}}\right)^{0.26}\left(\frac{\rho_{\mathrm{c}}^2\sigma^3}{\mu_{\mathrm{c}}^4\Delta\rho g}\right)^{0.17}$
Kumar and Hartland [57]	$\frac{Sh_{\mathrm{c}}/(1-\phi)-Sh_{\mathrm{c,rigid}}}{Sh_{\mathrm{c},\infty}-Sh_{\mathrm{c}}/(1-\phi)} = 5.26\times 10^{-2}\ Re^{1/3+6.59\times 10^{-2}\ Re^{1/4}}Sc_{\mathrm{c}}^{1/3}\left(\frac{V_{\mathrm{slip}}\mu_{\mathrm{c}}}{\sigma}\right)^{1/3}\frac{1}{1+\kappa^{1.1}}\left[1+C_1\left\{\frac{\psi}{g}\left(\frac{\rho_{\mathrm{c}}}{g\sigma}\right)^{1/4}\right\}^{n_1}\right]$	$C_1=C_2=4.33$ and $n_1=n_2=1/3$
	$Sh_{\mathrm{d}} = 17.7 + \frac{3.19\times 10^{-3}(Re\,Sc_{\mathrm{d}}^{1/3})^{1.7}}{1+1.43\times 10^{-2}(\mathrm{Re}\,Sc_{\mathrm{d}}^{1/3})^{0.7}}\left(\frac{\rho_{\mathrm{d}}}{\rho_{\mathrm{c}}}\right)^{2/3}\frac{1}{1+\kappa^{2/3}}\left[1+C_2\left\{\frac{\psi}{g}\left(\frac{\rho_{\mathrm{c}}}{g\sigma}\right)^{1/4}\right\}^{n_2}\right]$	$Sh_{\mathrm{c},\infty} = 50 + \frac{2}{\sqrt{\pi}}(Pe_{\mathrm{c}})^{1/2}$
	$Sh_{\mathrm{c,rigid}} = 2.43 + 0.775\ Re^{1/2}Sc_{\mathrm{c}}^{1/3} + 0.0103\ Re\,Sc_{\mathrm{c}}^{1/3}$ $k_i = Sh\,D_{\mathrm{AB}}/d$	
He et al. [46]	$K_{\mathrm{oc}}\underline{a} = 5.1705V_{\mathrm{c}}^{0.6685}Vd^{0.2404}(Af)^{0.5536}$, $D=0.6\,\mathrm{m}$	Correlation coefficient is sensitive to column diameter D. Constancy of exponents is questionable
	$K_{\mathrm{oc}}\underline{a} = 1.4296V_{\mathrm{c}}^{0.6685}Vd^{0.2404}(Af)^{0.5536}$, $D=0.05\,\mathrm{m}$ $K_{\mathrm{oc}}\underline{a} = 1.8125V_{\mathrm{c}}^{0.6685}Vd^{0.2404}(Af)^{0.5536}$, $D=0.1\,\mathrm{m}$	

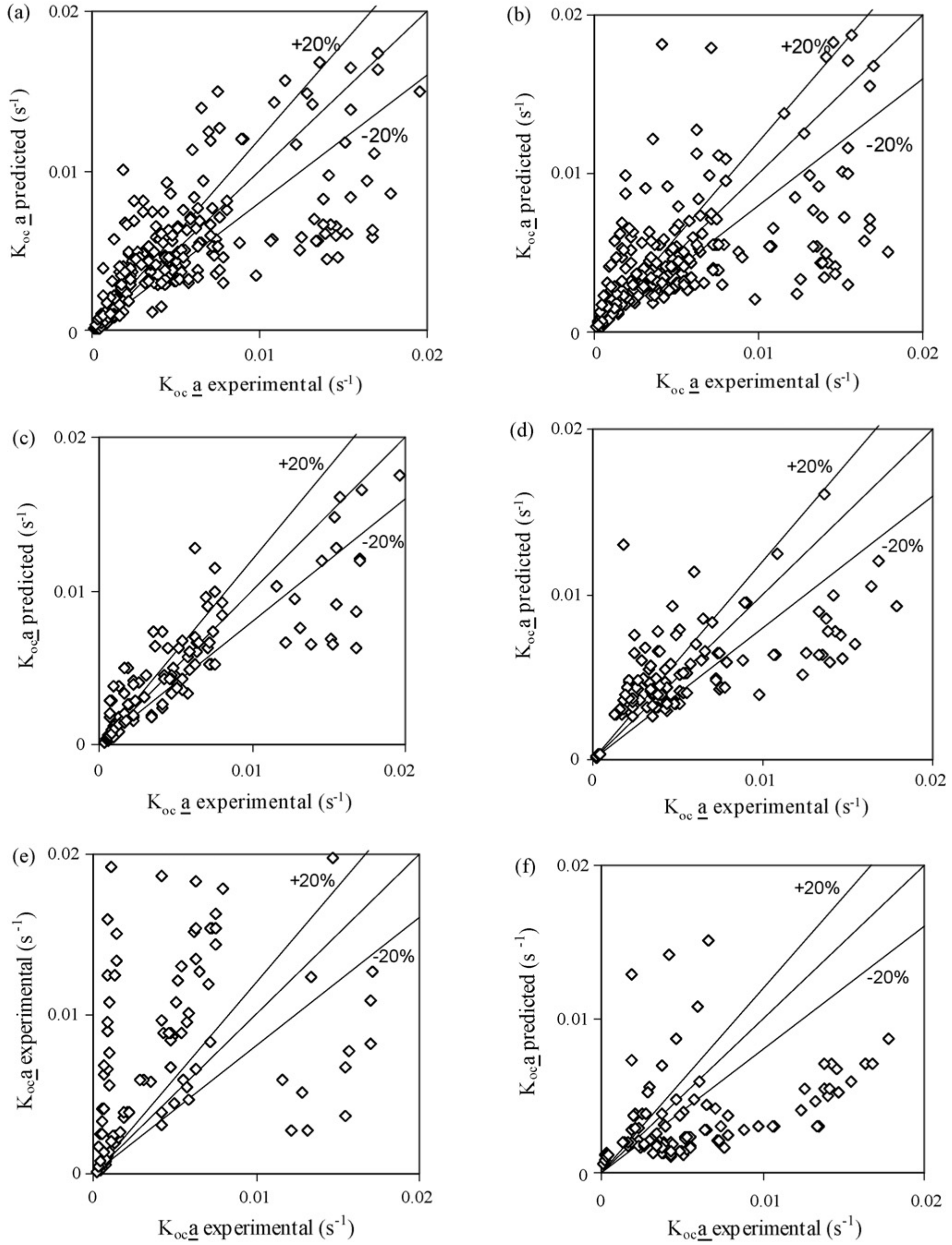

Fig. 7. Testing the predictive ability of different correlations for mass transfer coefficient in pulsed sieve plate columns: (a) Smoot et al. [4], (b) Thornton [13], (c) Smoot and Babb [52] (d), Smoot and Babb [52], (e) Kumar and Hartland [57], and (f) Kumar and Hartland [57]

experimental observation of various investigators [3,40]. Predictive ability of this correlation with the exponents obtained through regression is shown in Fig. 7a. Poor predictive ability can be related to the choice of dimensionless groups. The dimensionless groups used by reference [4] were based on the assumptions that column always operate in the emulsion regime and resistance to mass transfer lies in the dispersed phase film. Thus, these groups cannot be used for predicting the mass transfer coefficient for columns operating in other regimes. They also do not consider the effect of axial mixing on mass transfer efficiency of the column.

Thornton [13] has given another correlation for HTU_{oc} but it requires the knowledge of hold-up, and two constants (b and n) a priori. Experimental investigation is needed for finding the values of b and n. As a further step, the dimensionless groups in his correlation were taken and multiple regression technique was applied to it to calculate the exponents. Characteristic velocity (V_0) for their correlation was calculated using Eq. (5) as recommended by these authors. The hold-up required in this case was calculated using the slip velocity correlation of Venkatnarsaiah and Verma [45]. However, this approach was also found to be unsatisfactory as shown in Fig. 7b. Due to these discrepancies it was decided to separate the data in two categories depending on the direction of mass transfer. This resulted in 146 data points for mass transfer from continuous phase to dispersed phase (c → d) and 128 for mass transfer from dispersed phase to continuous phase (d → c). Smoot and Babb [52] have calculated the exponents in their correlation for d → c without taking into account the effect of interfacial tension. They also claimed that their correlation can predict HTUoc values for the liquid–liquid system MIBK–acetic acid–water with a deviation of 27%. However, when we compared their experimental values with those obtained from their correlation it was observed to give deviation of more than 40%. Further we applied the multiple regression technique to the correlation of reference [51] by including the term to account for the effect of interfacial tension. The predictive ability of this approach is shown in Fig. 7c. However, there was no improvement in the predictive ability of the correlation. The correlation of Venkatnarsaiah et al. [14] which gives the overall mass transfer coefficients was tried next. This correlation was found to give good result for the system for which it was developed. However, its predictability became poor when it was applied to liquid–liquid systems of varied physical properties. This could be because of the limited amount of the data with which this correlation was developed. Correlation of Luo et al. [54] was not tried since it was developed only for TBP–nitric acid–water system. Thus, none of the correlation was found to be satisfactory. Correlations of Smoot et al. [4] and Thornton [13] were tested for the mass transfer from c → d case. The predictive ability of the former correlation is shown in Fig. 7d. Exponents were obtained through regression analysis and the dimensionless groups (Reynolds Number, etc.) were now based on continuous phase. However, it was observed that in this case again the equations show poor predictability. Hence, it was concluded that none of the correlation present in the literature are general enough to predict the HTUoc values required for design of the column.

As a further attempt, it was decided to calculate the film mass transfer coefficients (k_c and k_d) and calculate the HTUoc values through this route. Interfacial area was calculated using $\underset{\sim}{a} = 6\phi/d_{32}$. Only one study from Kumar and Hartland [57] has attempted to give the film Sherwood number for pulsed columns. The film mass transfer coefficients were calculated from it and were used along with interfacial area and equilibrium constant (m) to calculate HTUoc. Fig. 7e and f show the predictive ability of this approach for mass transfer coefficients for mass transfer from d → c and c → d, respectively. However, a large deviation between the experimental and predicted values was obtained. Tang et al. [64] have used the correlation of Handlos and Baron [59] and Garner and Tayeban [65] cited from Tang et al. [64] for calculating k_d and k_c, respectively, for TBP–nitric acid–water system. When these equations of film mass transfer coefficients were used for calculating the HTUoc, considerable difference between the experimental and predicted values were obtained. Thus, none of the correlations presented in the literature were found to be applicable beyond the set of data for which they were formulated.

The design of an extraction column for a given separation warrants the availability of reliable correlations for the prediction of mass transfer coefficients. However, the value of $K_{oc}\underset{\sim}{a}$ varies with column geometry and physical systems. Mass transfer is a complex phenomenon and presence of even traces of impurities in the liquid–liquid system has a strong effect on it as compared to other parameter, viz. drop size, hold-up. Hence, unless each case is studied carefully and film transfer units evaluated, the application of correlations obtained for one system are not advisable to other systems and to other scales of operation. Therefore, it is difficult to obtain a generalized correlation for mass transfer. It is therefore recommended to undertake pilot scale experiments to find out mass transfer rates for actual industrial systems.

3. Conclusions

Design aspects of the pulsed sieve plate extraction columns have been studied. Numerous correlations pertaining to hydrodynamics and mass transfer characteristics of these columns have been evaluated. Applicability of different correlations was tested against a large set of experimental data gathered from the work of different investigators over the past 50 years. Since flooding tendency of the column decides its diameter, predictive ability of different correlations for flooding in the pulsed sieve plate extraction column was adjudged. It was found that the correlation of Tribess and Brunello [19], which relates the maximum in the flooding curve to geometrical and operating characteristics of the column and physical properties of liquid–liquid systems, has good predictive ability. It was able to predict the maximum in the flooding curve for all the systems recommended by EFCE. All correlations of Kumar and Hartland [27,33,34] were found to overpredict the drop size. This could be related to larger exponent on interfacial tension (0.6) and absence of hole diameter in all the correlations. Correlation of Srinivasulu et al. [35] was found to predict the drop size satisfactorily. Both the major empirical correlations for direct calculation of hold-up [7,45] were found to overpredict it value. A separate approach of calculating hold-up through a correlation for slip velocity (using Eq. (4)) was followed. It was found that hold-up obtained using the V_{slip} correlation of Venkatnarsaiah and Verma [45] is in good agreement with the corresponding experimental value. Hence, the V_{slip} correlation [45] is recommended for predicting hold-up in the pulsed sieve plate extraction columns. For mass transfer, none of the correlations were found to be satisfactory and hence pilot scale experiments are recommended. Thus, the exercise has helped in identifying a correlation each for flooding, drop size and hold-up which can be used for design of pulsed columns. The results of the present study can reduce the experimental work associated with the design of the pulsed columns and their scale-up.

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