Determination of Expansion Properties and Liquid Mixing in Expanded Bed: A simple unique expanded bed contactor (NBG expanded bed column with internal diameter 0.013 and 0.25 m in height), which was equipped with a glass tube fitted with a simple sintered glass distributor comprising a 40 μm mesh, has been prepared and used herein [9, 10]. To regulate the position of the liquid outlet to the top of the expanded bed, a movable adapter was used. Normally the adsorbent particles were added to 0.06 m [11] sedimented bed height. In order to transport the fluid, a peristaltic pump (B-V Trat Acque, Etatron DS-Rome, Italy) was used. Proper column vertical alignment was positioned in all experiments. The experiments were carried out in deionized water at 25°C. The degree of the expanded bed was measured three times for each flow velocity after the equilibrium expansion for 30min. Then the mean value of degree of the expanded bed was used to estimate the bed expansion factor and bed viodage [12].

$$E = \frac{H}{H_0} \tag{5}$$

Where H and H_0 represent the expanded bed height and the sedimented bed height, respectively. In this study, H_0 was 6 cm. To describe the expansion properties, the well-known Richardson-Zaki equitation was used as follows [13].

$$U = U_t \varepsilon^n \tag{7}$$

Based on relation between the voidage of expanded bed (ε) with the superficial liquid velocity (U), the terminal settling velocity of particle (U_t) and the expansion index (n) could be calculated. Bed voidage was calculated by Eq. 7.

$$E = \frac{H}{H_0} = \frac{1-\varepsilon_0}{1-\varepsilon} \tag{7}$$

Where, the ratio of expanded bed height (H) to sediment bed height (H_0) is named the expansion factor (E) and the voidage of sedimented bed (ε_0) is commonly supposed as 0.4 [14].

Then, according to Eqs. (3) and (4), experimental values of U_t and n can be determined by the double logarithmic plot of ε vs. U.

Residence Time Distribution (RTD) measurements were performed using a negative step signal method. A bed of the adsorbent particles was fully expanded using buffer A at tested flow rates. A dilute acetone solution (10% v/v) was used as the input to the column in a system fluidised with buffer A. The UV absorbance of the acetone was measured by spectrophotometer (Jenway 6305 UV/VIS. spectrophotometer, Germany) at wave length of 280 nm [15] in stream output from the column using a UV monitor. The Bodenstein number (B_o) and axial dispersion coefficient (D_{ax}) which express the state of liquid dispersion and fluidisation behaviour, were calculated by the following equations [8]:

$$N = 5.54\left(\frac{t_R}{W_{1/2}}\right)^2 \tag{8}$$

Where t_R is the residence time and $W_{1/2}$ is the half peak width.

Eq. 9 calculates value of HETP as follows:

$$HETP = \frac{H}{N} \tag{9}$$

Equation below defines the B_o number relating convective transport of liquid to dispersion:

$$B_0 = \frac{UH}{D_{ax}\varepsilon} \tag{10}$$

In addition, Eq. 11 calculates B_o number as follows:

$$\frac{1}{N} = \frac{2}{B_0} + \frac{8}{B_0^2} \tag{11}$$

Thus, D_{ax} is defined by Eq. 7.

RESULTS AND DISCUSSIONS

Particle Appearance and Physical Characteristics: Figure 1 shows the morphology of the prepared matrix under the optical microscope. The prepared matrix was in a uniform spherical shape and no fragments were observed. The important physical properties of expanded bed matrices are considered, including sphericity, wet density, water content, size distribution and pore construction [16]. In the present work, Streamline DEAE was used for comparison purposes. The main physical properties of Agarose-Nickel matrix and Streamline DEAE were measured and listed in Table 1.

The size distribution of prepared matrix was measured and the results are illustrated in Figure 2. Using the size distribution data, the size range and mean particle size were investigated and listed in Table 1. Agarose-Nickel particles have the logarithmic symmetrical distribution. The mean size of Agarose-Nickel particles is about 126.4 μm, which is smaller than Streamline DEAE (203 μm).

Table 1: The physical properties of the Agarose-Nickel matrices and Streamline DEAE

Matrix	Size range(µm)	Mean size(µm)	ρ_p (g/ml)	ω (%)	P (%)
Agarose-Nickel	50-200	126.4	1.95	49.5	91.93
Streamline DEAE [17]	100-300	203	1.16	74.3	86.4

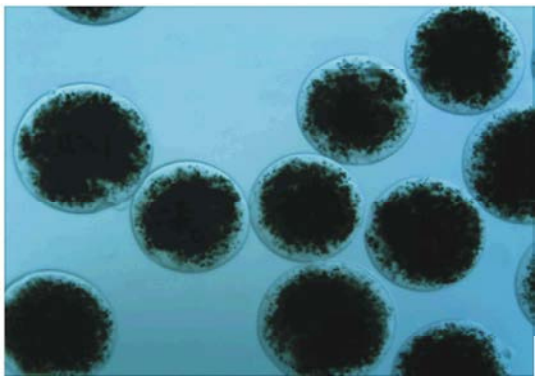

Fig. 1: Optical micrograph of Agarose-Nickel matrix

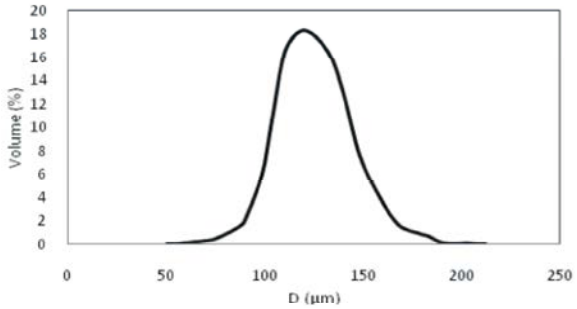

Fig. 2: Size distribution of Agarose-Nickel matrix

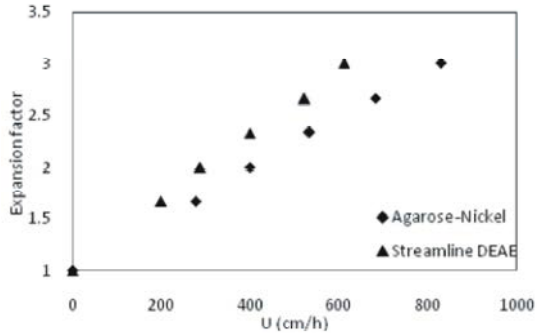

Fig. 3: Bed expansion for Agarose-Nickel matrix and Streamline DEAE

Generally, wet density of adsorbent plays an important role in performance of expanded bed [8]. Wet density of matrix determines the operation velocity of the expanded bed and high matrix density means high productivity of the separation process [5].

Based on physical properties of the Agarose-Nickel matrices and Streamline DEAE (Table 1), the wet density of prepared matrix was 1.95 g/ml, which is significantly higher than that of Streamline DEAE bead (1.16 g/ml) [17]. Considering the differences of densities between Nickel powder (8.9 g/ml) and quartz (2.4 g/ml), addition of Nickel powder at the same volume ratio produced a heavier matrix. The results indicated that prepared matrix can be used at higher flow velocity.

Hydrodynamic Characteristics

Expansion Characteristics and Correlations: The bed expansion contributes to the adsorption efficiency as a composite function of liquid distribution, liquid properties (e.g. viscosity, density and content of cell homogenate), particle characteristics and the configuration of the column in terms of wall and distributor effects [15]. The expansion characteristics of Agarose-Nickel matrix and Streamline DEAE were measured under the normal operating conditions for EBA. The results are shown in Figure 3. Obviously big and heavy particles caused low expansion at the same fluid velocity [18]. The Streamline DEAE matrices have shown bigger particle diameter and smaller wet density in compare to the prepared matrices of present work (Table 1). It can be deduced from Figure 3 that a matrix having a low wet density does not necessarily exhibit a high degree of bed expansion at a given fluid velocity (compare the prepared matrix and Streamline DEAE degree of bed expansion at the different values of fluid velocity) [15]. It meant that bed expansion of Agarose-Nickel matrix was lower than that of Streamline DEAE at the same flow velocity.

The perfect operation expansion factor of EBA is in the range of 2-3 and Agarose-Nickel matrix could be used for a fluid velocity of 398.72 to 829.77 cm/h and Streamline DEAE for a fluid velocity of 286.55 to 611.23 cm/h. Therefore, Agarose-Nickel matrix is beneficial for the separation with fast adsorption and high productivity.

The famous Richardson-Zaki equation (Eq. 6) has been used to correlate the expansion factor and bed voidage. It was observed that Richardson-Zaki equation could describe the relationship between the expansion voidage and the flow velocity for the prepared matrix and Streamline DEAE (Figure 4). The decision coefficients (R^2) were 0.992 for the prepared matrix and Streamline DEAE.

The correlated parameters are listed in Table 2. The expansion index (n) was 4.9 for the prepared matrices and 5.12 for Streamline DEAE matrices and consequently near to the theoretical value of 4.8 in the laminar flow

Table 2: Parameters of U_t and with Richardson-Zaki equation.

Matrix	U_t (cm/h)	n
Agarose-Nickel	2377.96	4.9
Streamline DEAE	1878.07	5.12

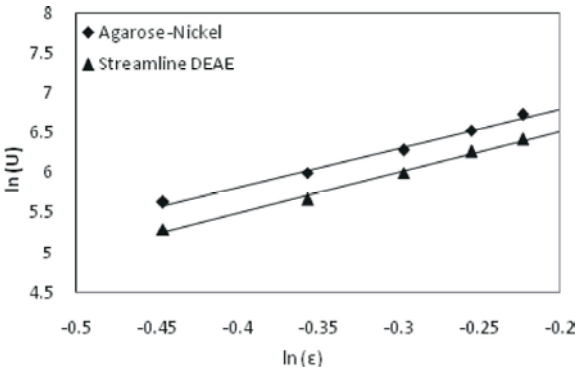

Fig. 4: Richardson-Zaki correlation between flow velocity and bed viodage

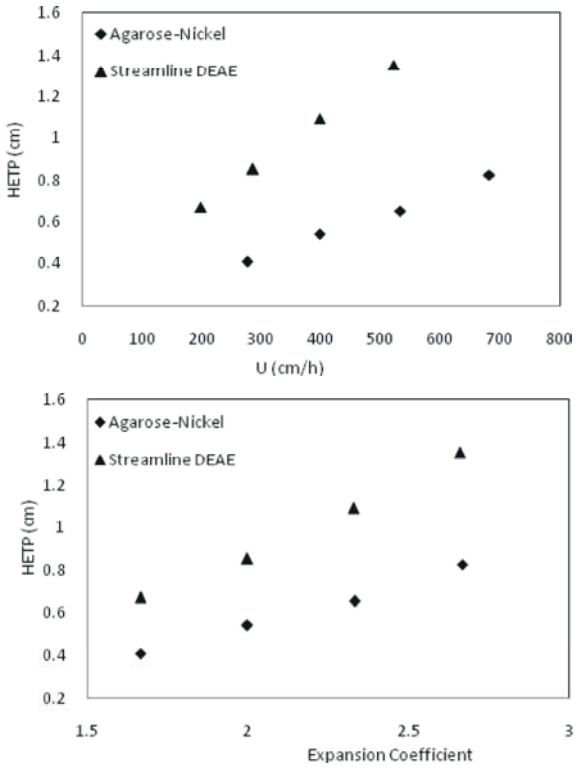

Fig. 5: HETP as the function of flow velocity and bed expansion

regime. In addition, it was observed that terminal settling velocity (U_t) of Agarose-Nickel n correlated matrix was higher than that of Streamline DEAE.

Mixing of the Liquid Phase in the Expanded Bed: The fluidization stability of adsorbent in the expanded bed is another important factor, which should be considered

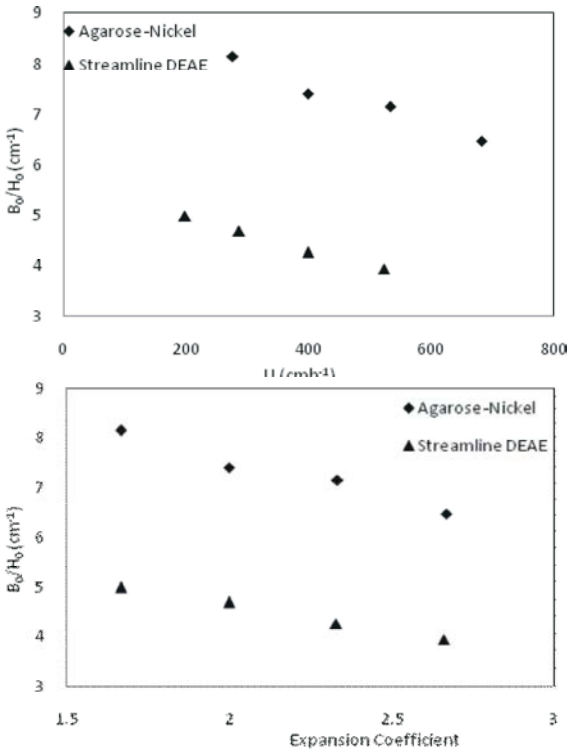

Fig. 6: B_0/H_0 number as the function of flow velocity and bed expansion

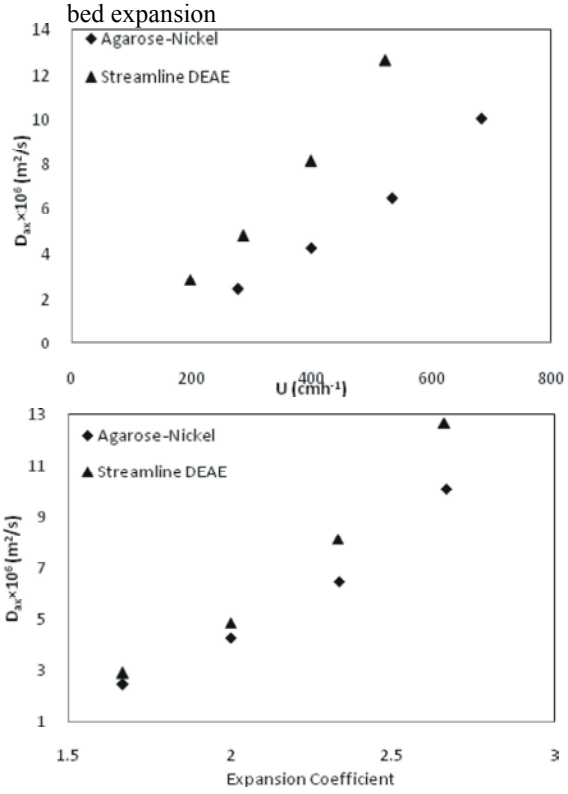

Fig. 7: Axial mixing coefficient (D_{ax}) as the function of flow velocity and bed expansion

in the EBA applications [20]. Measurement of the axial dispersion, i.e. the deviation from plug flow movement of fluid elements in an adsorbent bed (both packed and fluidized), is commonly performed by RTD analysis of step or pulse signals [11]. In this work, this method was employed in order to characterize and compare Agarose-Nickel matrix with a commercial matrix. To investigate the characteristic of liquid mixing in the column, the height equivalent of a theoretical plate (HETP), Bodenstein number (*Bo*) and the axial mixing coefficient (D_{ax}) are used.

As noticed in Eqs. (8) and (9), the number of theoretical plates (N) and then the height equivalent of theoretical plate for both the matrices under corresponding operation conditions were calculated. Figure 5 depicts HETP as the function of flow velocity and bed expansion. The prepared matrices showed smaller HETP than commercial matrix.

To calculate the B_0 numbers, Eq.11 was used. To eliminate the effect of different initial bed height, the parameter of B_0/H_0 was used [21]. Figure 6 shows the B_o/H_0 as the function of expansion factor and flow velocity. The values of B_0/H_0 ranged from 6.46 to 8.15 cm^{-1} for Agarose-Nickel matrix and 3.94 to 4.98 cm^{-1} for Streamline DEAE. It meant that the expanded bed with Agarose-Nickel composite matrix is more stable than that with Streamline DEAE.

The axial mixing coefficients (D_{ax}) were calculated in Eq. (10) and the results are shown in Figure 7. It was observed that under corresponding operation conditions, the D_{axl} values of the prepared matrix were lower than that of Streamline DEAE, which indicated that the prepared matrix had better hydrodynamic properties than commercial matrix. In this study, at fluid velocity of 276.49-829.77 cm/h, the D_{ax} values of the prepared matrix were between 1.0×10^{-6} and 1.0×10^{-5} $m^2.s^{-1}$. According to the literature, this range of the D_{ax} values will benefit EBA application [17]. In addition, for a normal operation velocity of 520 cm/h, the values of D_{ax} were lower than 6.5×10^{-6} $m^2.s^{-1}$ for the prepared matrix and 12.6×10^{-6} $m^2.s^{-1}$ for Streamline DEAE. The results indicated that Agarose-Nickel matrix can formed a better stable expanded bed than Streamline DEAE.

As illustrated in Figures 5, 6 and 7, with increasing fluid velocity, the HETP values increased, the B_o values decreased and the D_{ax} obviously increased. It indicated that the stability of expanded bed would be disturbed with the increase of flow rate.

CONCLUSION

A novel type of composite matrix was prepared with Nickel powder as the densifier and Agarose as the skeleton using the method of water-in-oil emulsification. The physical properties of Agarose-Nickel matrix were measured. The prepared matrix had the spherical appearance, suitable size and size distribution, appropriate wet density of 1.95 g/ml and porosity of 91.93%. The bed expansion factor in the range of 2-3 was demonstrated and correlated with Richardson-Zaki equation. In addition, the theoretical prediction of correlation parameters (the terminal settling velocity (U_t) and expansion index (n) was measured. The stability of expanded bed was studied by residence time distribution (RTD) experiments and the common quantities used to describe the expanded bed process were discussed (i.e. height equivalent of a theoretical plate (*HETP*), Bodenstein number (B_o) and axial dispersion coefficient (D_{ax}). The obtained results indicated that Agarose-Nickel matrix shown better feature of expansion, lower axial mixing than the commercial Streamline DEAE matrix. Furthermore, the results indicated that the stability of expanded bed decreased with the increase of fluid velocity.

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Notation:

B_o = Bodenstein number dimensionless
D_{ax} = Axial dispersion coefficient m^2/s
E = Bed expansion factor dimensionless
H = Height of expanded bed cm
H_0 = Height of sedimented bed cm
M_1 = Mass of wet matrix g
M_2 = Mass of a gravity bottle filled with water g
M_3 = Mass of a gravity bottle with wet matrix full of water g
m_1 = Mass of weighing bottle g
m_2 = Mass of matrix and weighing bottle before drying g
m_3 = The mass of matrix and weighing bottle after drying g
n = Richardson-Zakai parameter dimensionless

N = The number of theoretical plates dimensionless
P = Porosity %
t_R = Retention time min
U = Superficial fluid velocity cm/h
u_t = Terminal velocity of a single adsorbent particle in a volume of stagnant liquid cm/h
$W_{1/2}$ = Half-peak width min
V = Pore volume ml/g
$HETP$ = The height equivalent of theoretical plate dimensionless

Greek Letter:

ρ_c = Wet density of the particles g/ml
ρ_W = Density of water g/ml
ω = Water content %
ε = Voidage of expanded bed dimensionless
ε_0 = Voidage of sedimented bed dimensionless

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