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APPENDICES

Appendix A: Crystal size data from SEM analysis

Table A1 Crystal size data of Molecular Sieve Zeolite of size 1/8"

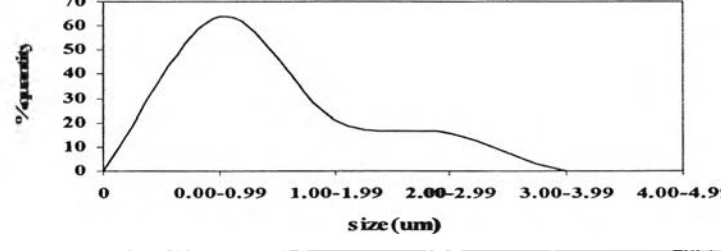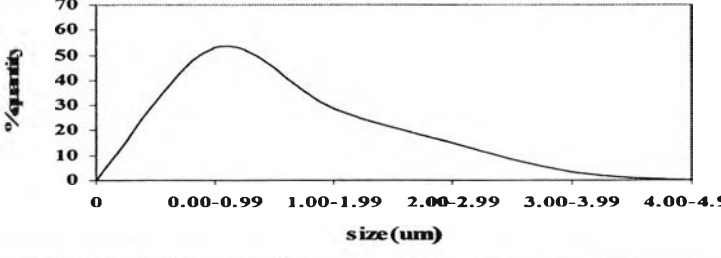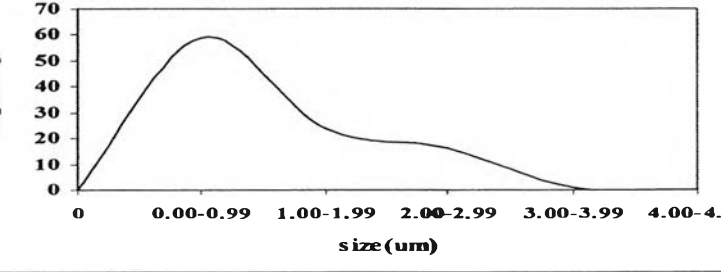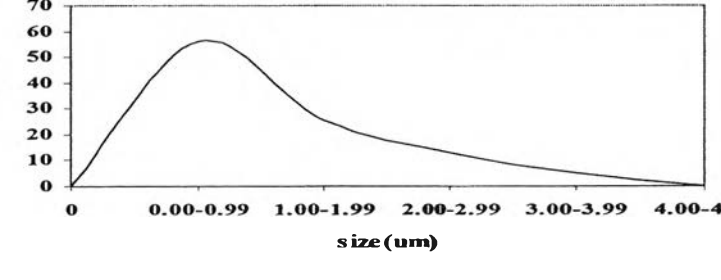| Crystal size range (micron) / Aging condition	%quantity				Average crystal size (micron)
	0.00-0.99	1.00-1.99	2.00-2.99	3.00-3.99	
Fresh	0.96	34.62	46.15	18.27	2.312
10 batches	4.21	47.37	37.89	10.53	2.042
20 batches	8.15	48.91	40.76	2.17	1.865
30 batches	14.04	52.63	24.56	8.77	1.776
40 batches	21.70	39.62	37.74	0.94	1.531
50 batches	51.90	34.06	13.54	1.31	1.271
60 batches	50.00	35.09	12.28	2.63	1.170
70 batches	63.97	20.65	15.38	0.00	1.168
80 batches	52.91	28.64	15.05	0.00	1.134
90 batches	58.74	23.79	16.50	0.00	1.104
100 batches	56.10	25.61	13.01	0.00	1.098
120 batches	63.40	22.50	12.60	0.00	1.064
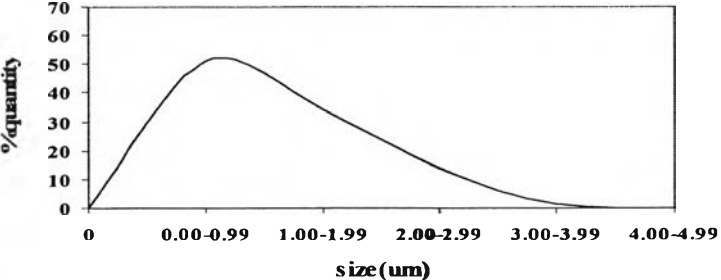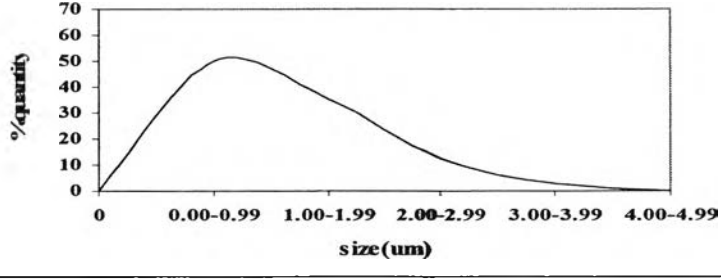
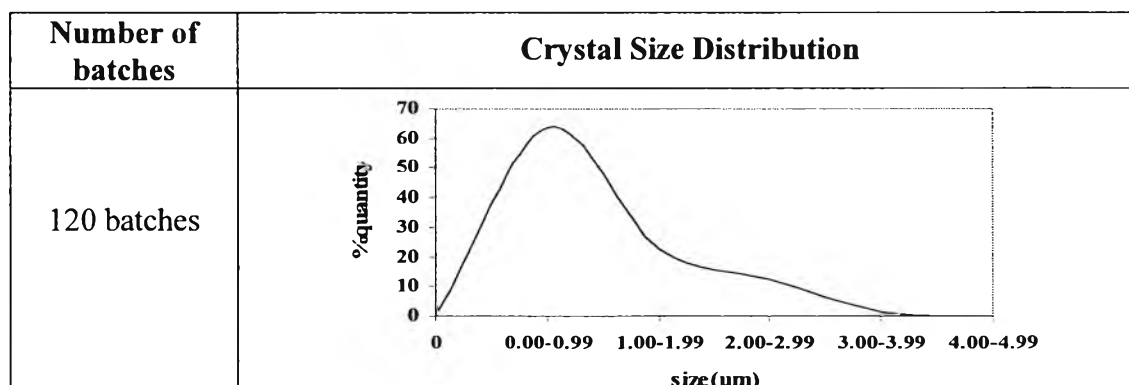**Table A2** Crystal size data of Molecular Sieve Zeolite of size 1/16"

Crystal size range (micron) / Aging condition	%quantity				Average crystal size (micron)
	0.00-0.99	1.00-1.99	2.00-2.99	3.00-3.99	
Fresh	12.68	33.80	33.40	19.72	2.101
10 batches	10.24	36.22	37.80	15.75	2.086
20 batches	9.21	47.37	26.32	17.11	2.008
30 batches	31.64	28.91	25.78	13.67	1.722
40 batches	34.21	30.53	22.63	12.63	1.653
50 batches	32.58	31.67	27.60	7.24	1.617
60 batches	42.97	36.88	14.45	5.70	1.328
70 batches	51.16	37.79	9.30	1.74	1.111
80 batches	54.74	32.28	11.23	1.75	1.095
90 batches	59.87	29.15	8.78	2.19	1.028
100 batches	65.38	20.94	11.54	2.14	0.999
120 batches	66.40	22.00	10.50	1.10	0.982

**Appendix B: Crystal Size Distribution of Molecular Sieve Zeolite of size 1/8"
and 1/16"**

Table B1 Crystal Size Distribution of Molecular Sieve Zeolite of size 1/8"

Number of batches	Crystal Size Distribution
Fresh	
10 batches	
20 batches	
30 batches	
40 batches	
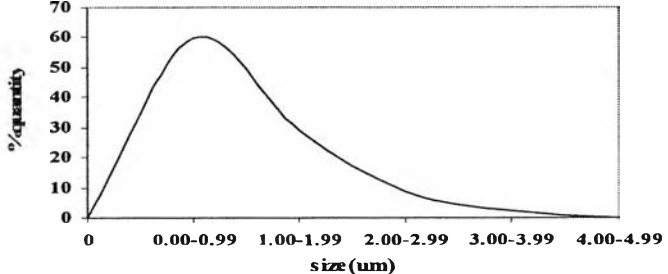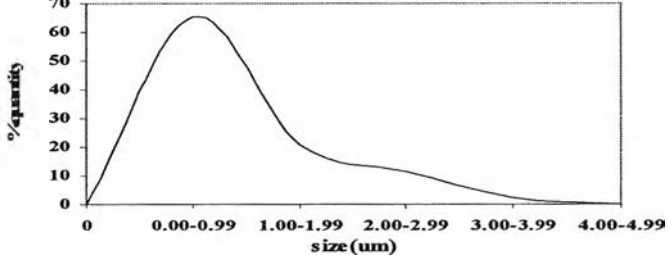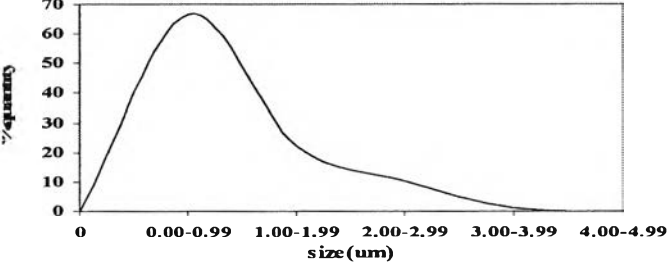
Number of batches	Crystal Size Distribution
50 batches	
60 batches	
70 batches	
80 batches	
90 batches	
100 batches	

Number of batches	Crystal Size Distribution
120 batches	

Table B2 Crystal Size Distribution of Molecular Sieve Zeolite of size 1/16"

Number of batches	Crystal Size Distribution
Fresh	
10 batches	
20 batches	
30 batches	
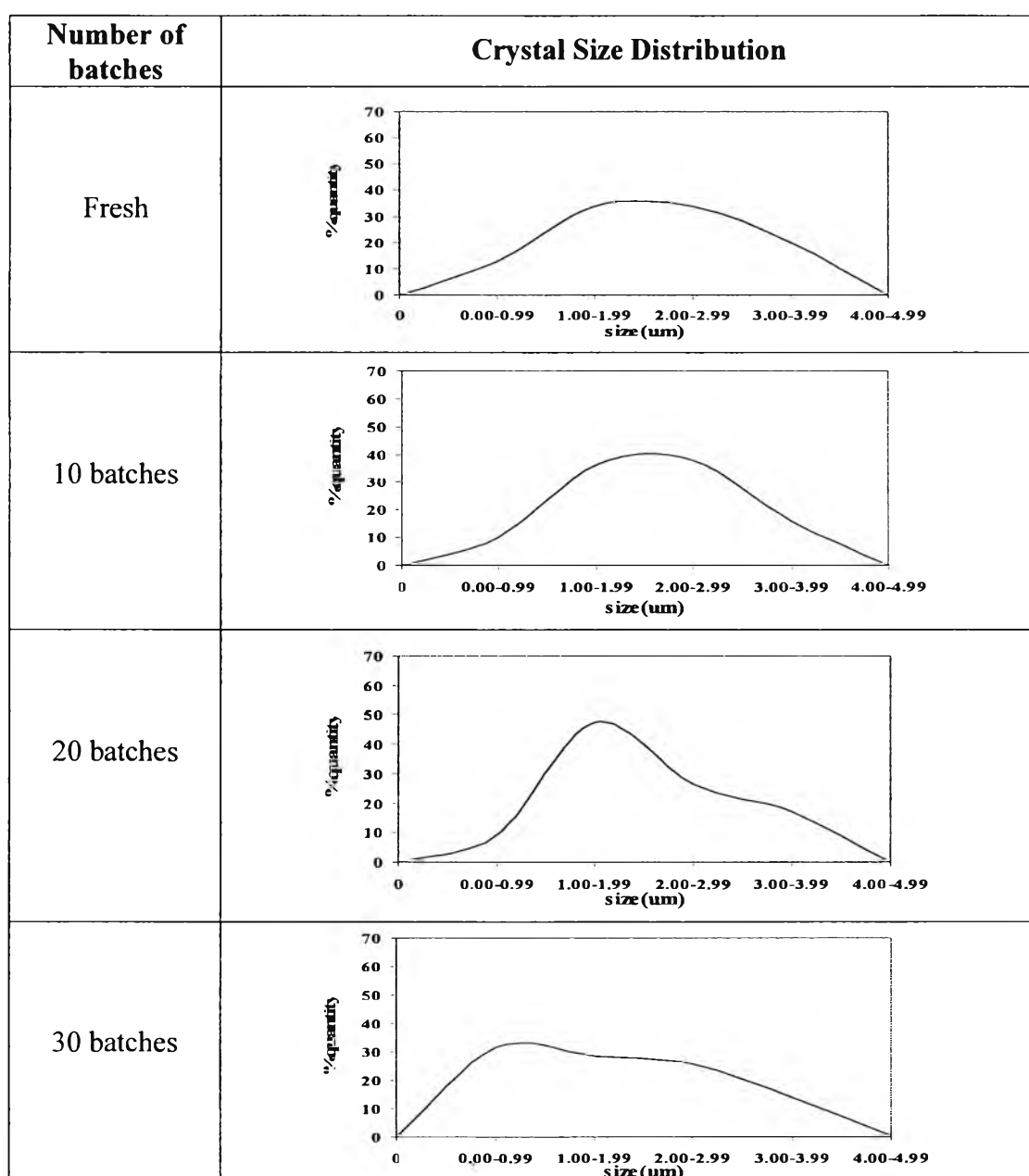
Number of batches	Crystal Size Distribution
40 batches	
50 batches	
60 batches	
70 batches	
80 batches	

Number of batches	Crystal Size Distribution
90 batches	
100 batches	
120 batches	

Appendix C: Conditions for Adsorption experiments

Table C Conditions for Adsorption Experiments

Parameters	Value
Weight of adsorbent packed in adsorber	about 5 g
Operating pressure (const.)	1 atm
Operating temperature (isothermal)	25°C
Humidity of natural gas feed	5-75%RH
Natural gas feed flow rate	
Activated alumina	115.1 ml/min
Molsiv (1/8")	125.8 ml/min
Molsiv (1/16")	138.8 ml/min
Contact time(based on feed flow rate)	
Activated alumina	11.83 sec
Molsiv (1/8")	10.82 sec
Molsiv (1/16")	9.81 sec
Bed volume	24.3 ml

Appendix D: Conditions for breakthrough curve experiments

Table D Multi-layer adsorber

Adsorber layout	Adsorbent type	Height (cm)	Actual volume ratio* (%)
Bed volume: 75ml	Activated alumina	0.3	3.41
	MolSiv Zeolite type 4A size 1/8"	5.6	63.64
	MolSiv Zeolite type 4A size 1/16"	2.9	32.95
	Ceramic ball	Inert material used as an adsorbent support	

*PTT Public Co.Ltd.

Appendix E: Parameters Applied in the Mathematical Model

E.1 Parameters Applied in the Mathematical Model

Table E1 Parameters applied in the mathematical model

Parameter	alumina	1/8" 4A Molsiv	1/16" 4A Molsiv
Bed void fraction*, ε	0.37	0.35	0.34
Axial dispersion coefficient**, D_L	0.02824	0.027733	0.027170
Bulk density***, (g/cm^3)	0.73743	0.66529	0.66529

* Appendix F
** Appendix E.3
*** UOP information

E.2 Interstitial velocity

Since the interstitial velocity can be calculated from the correlation, $v_i = v_s / \varepsilon$, so the superficial velocity and bed void fraction are needed.

- Superficial velocity

$$v_s = \frac{Q}{A}$$

Where, Q = 457.78 ml/min

A = cross sectional area of adsorber = 8.55 cm

So, v_s = 53.52 ml/min

- Interstitial velocity

$$v_i = \frac{v_s}{\varepsilon}$$

	ε	v_i (ml/min)	contact time(sec)
alumina	0.37	144.65	0.964
molsiv(1/8")	0.35	152.91	18.73
molsiv(1/16")	0.34	157.41	9.42
		all bed	9.83

E.3 Dispersion Coefficient

- Molecular diffusion coefficient, D_m is calculated from Chapman-Enskog equation.

$$D_m = \frac{0.0018583 T^{3/2} (1/M_A + 1/M_B)^{1/2}}{P * \sigma_{AB}^2 \Omega_{AB}}$$

Where, M_A = average molecular weight of bulk species (natural gas) = 20.59

M_B = molecular weight of adsorbate species (water) = 18

σ_{AB} = collision diameter from Lennard-Jones potential = 3.29352 °A

Ω_{AB} = collision integral = 1.60089

P = total pressure in atmospheres = 1 atm

T = temperature = 298 K

Appendix F: Bed void fraction of each adsorbent

The bed void fraction of the adsorbents used in the packed bed adsorber can be obtained from Figure F1.
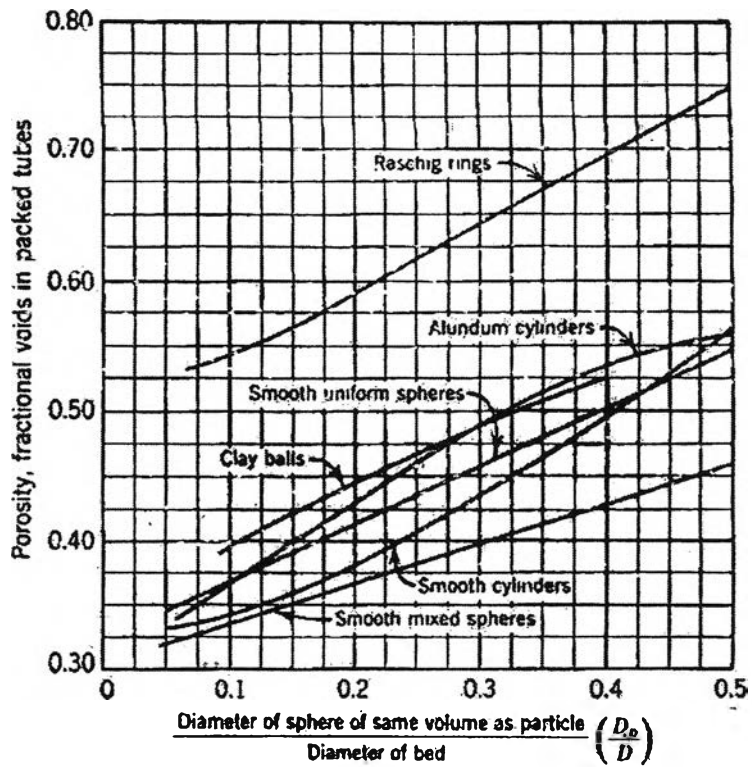
Figure F The porosity as a function of the ratio of particle diameter to bed diameter (Leva, 1947).

Appendix G: Sensitivity of Overall Mass Transfer Coefficient (k) to the Shape of the Breakthrough Curve

Uttamaroop (2003) indicated the increase of overall mass transfer coefficient resulted in steeper breakthrough curve, and then the k value of about 8.5×10^{-5} s^{-1} was suggested. But in this work, the result obtained from the slope of theoretical breakthrough curve using $k = 8.5 \times 10^{-5}$ s^{-1} was quite different from the experimental one as shown in Figure G1. So, the breakthrough curve from using the k value of about 5.0×10^{-5} s^{-1} was examined, and it was found the breakthrough pattern was similar to the experimental one. But the breakthrough time predicted from the model of $k = 8.5 \times 10^{-5}$ s^{-1} was more precise, with 6.47% error from the experiment. The difference of errors is shown in Table G1. Thus, the breakthrough curve using $k = 8.5 \times 10^{-5}$ s^{-1} was employed for good agreement with the experimental data under identical conditions.
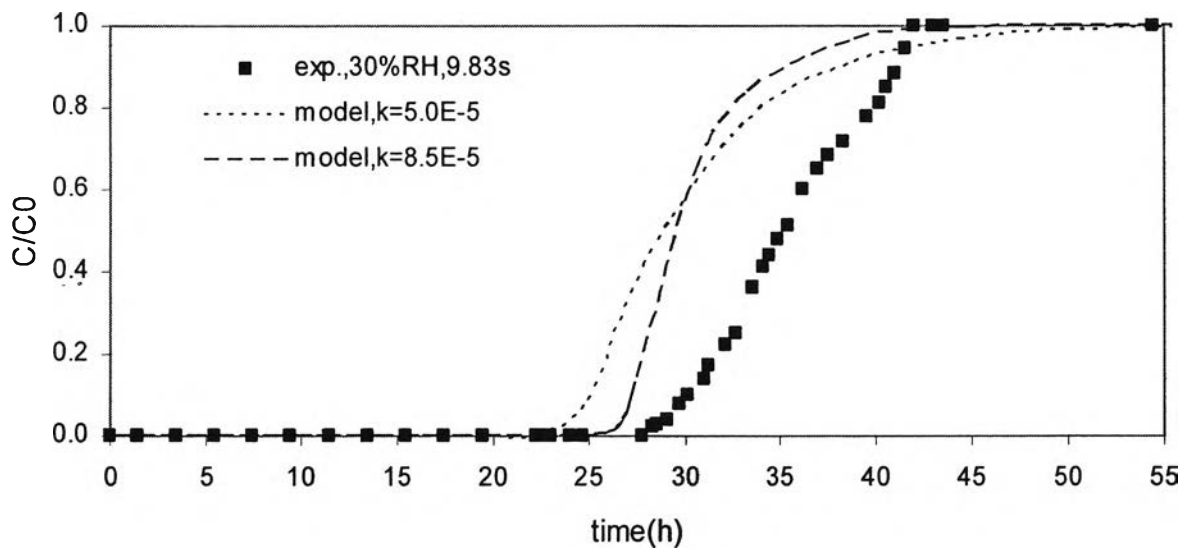
Figure G   Comparison between the experimental and theoretical breakthrough curves with the influence of the overall mass transfer coefficient at the contact time of 9.83 sec and the feed humidity of 30%RH.

Table G Comparison of breakthrough time predicted from several k value

k value	Theoretical breakthrough time(h)	% Error
5.0 E-05	25	10.07
8.5 E-05	26	6.47

Appendix H: Effect of the Bed Voidage on the Breakthrough Time of Deactivated Adsorbents

The equilibrium adsorption isotherm equations of deactivated adsorbents were applied in the mathematical model to predict the breakthrough profile at the same conditions. From the model, the breakthrough occurs at around 19 hours, resulting in about 20.83 % error from the experiment. Therefore, another parameter related to the adsorbent deactivation inside the packed bed, which is bed voidage, was studied.

The void fraction of deactivated adsorbents were corrected based on the sensitivity analysis of Uttamaroop (2003), which suggested that the theoretical breakthrough curve shifted to the longer time when the bed void fraction decreased. This suggestion was supported with the result from the adsorbents characterization part which when the molecular sieve zeolite were deactivated, the pellets were destroyed, and their size were decreased. Thus, the corrected void fractions for both molecular sieves were applied in the model and gave the good agreement with the experimental data. New void fractions were measured and calculated using data from Appendix F and shown in Table H1. The new breakthrough time was around 22 hours and gave about 8.3% error.

Table H The bed void fraction provided in the theoretical breakthrough model for 88.3% deactivated alumina, 15.13% deactivated 1/8" molsiv, and 14.10% deactivated molsiv at the contact time of 9.83 sec and the feed humidity of 30%RH

Adsorbent	Void fraction	Corrected void fraction
alumina	0.37	0.37
1/8" molsiv	0.35	0.30
1/16" molsiv	0.34	0.29

In addition, another parameter that could affect the theoretical breakthrough curve when the void fraction changes is the axial dispersion coefficient because this parameter obtained from calculation by taking into account the void

fraction in calculation. But from sensitivity analysis by Uttamaroop (2003), it was found the theoretical breakthrough curve was not sensitive to the change of axial dispersion coefficient. So, the effect of this parameter was not studied in this work.
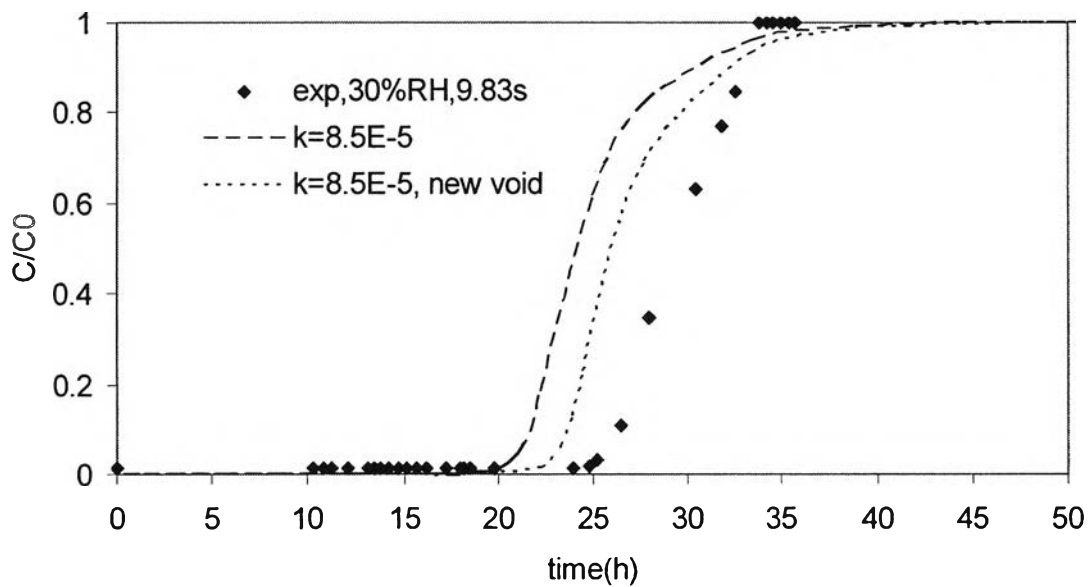
Figure H Comparison between the experimental and theoretical breakthrough curves of deactivated adsorbents at the contact time of 9.83 sec and the feed humidity of 30%RH.

Appendix I: Hydrothermal steaming apparatus
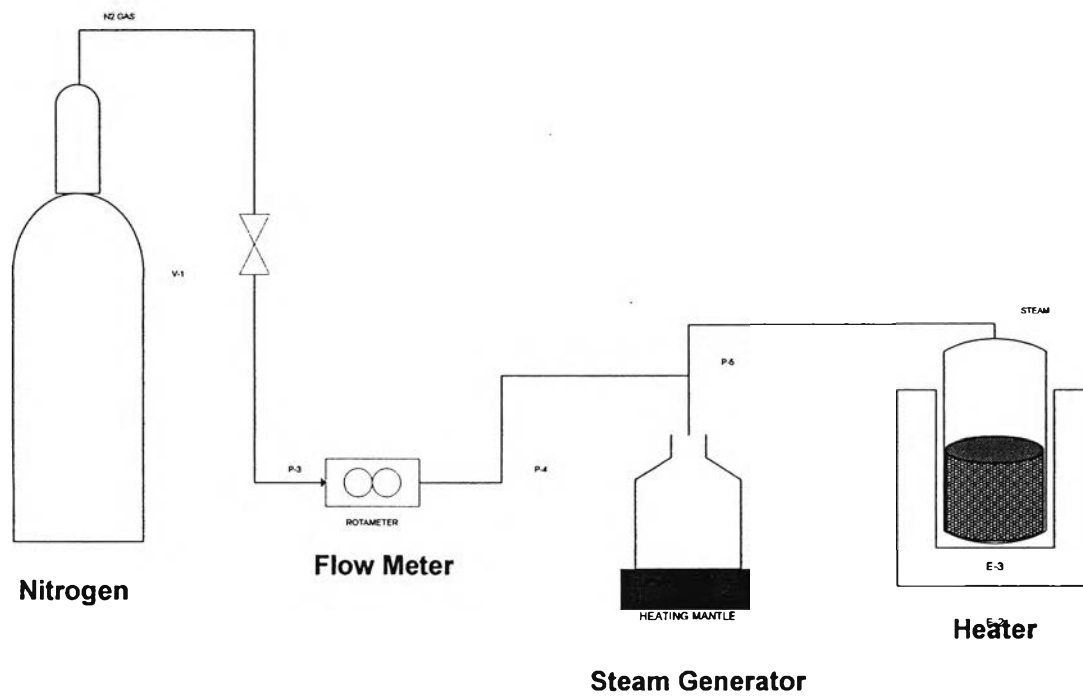
Figure I Hydrothermal steaming apparatus.

Appendix J: Simulation Program

```
PROGRAM BREAKTHROUGH CURVE PREDICTION

        ! for the assumption of velocity is not constant

        IMPLICIT NONE
        DOUBLE PRECISION C0,C,q,dt,KC,Kq,time,Csat,RH
        DOUBLE PRECISION v1,v,e_A,e_B,e_C
        INTEGER i,j,imax,jmax,n
        PARAMETER(imax=90,jmax=5000)
        DIMENSION C(imax,jmax),q(imax,jmax),v(imax,jmax)
        DIMENSION KC(4,imax,jmax),Kq(4,imax,jmax)

C       ...............INITIAL CONDITION................
        dt = 0.008
        time=0
        j=1                             !at time = 0
        RH = 30
        Csat=1.2794E-03
        C0 = RH*Csat/100       !inlet concentration (mol/l)
        C(1,j)=C0
        v1= 0.8920833          ! Superficial velocity
        e_A=0.37
        e_B=0.35
        e_C=0.34
        v(1,j)=v1/e_A

        OPEN(5,file='velocity.dat')

        DO i=2,imax

           C(i,j)=0
           q(i,j)=0

        END DO

        OPEN(1,file='data1.dat')

        DO i=1,imax
           WRITE(1,101)time,i,j,C(i,j),q(i,j),v(i,j)
        END DO

           Call RK4(j,C,q,v,imax,jmax,KC,Kq,dt)

        GOTO 20

10      OPEN(2,FILE='DATA2.DAT')

        DO i=2,imax

           READ(2,102)time,C(i,1),q(i,1),v(i,1),(KC(n,i,1),n=1,4)
        1    ,(Kq(n,i,1),n=1,4)

        END DO

20      DO j=2,jmax
```

```
      DO  i=2,imax

          v(1,j)=v1/e_A

          C(1,j)=C0

          C(i,j)=C(i,j-1)+(dt/6)*(KC(1,i,j-1)+2*KC(2,i,j-1)
     1            +2*KC(3,i,j-1)+KC(4,i,j-1))

          q(i,j)=q(i,j-1)+(dt/6)*(Kq(1,i,j-1)+2*Kq(2,i,j-1)
     1            +2*Kq(3,i,j-1)+Kq(4,i,j-1))

      END DO

      Call      RK4(j,C,q,v,imax,jmax,KC,Kq,dt)

      END DO
C      .............The Results...................

      DO j=1,jmax

          time=time+dt            !sec

      END DO

      CLOSE(2)
      OPEN(2,file='data2.dat')
      DO i=2,imax

          WRITE(2,102)time,C(i,jmax),q(i,jmax),v(i,jmax)
     1      ,(KC(n,i,jmax),n=1,4),(Kq(n,i,jmax),n=1,4)

          WRITE(5,102)time,C(i,jmax),q(i,jmax),v(i,jmax)

          OPEN(3,file='FFF_NEW_30%RH&9.83sec(vnotconst).dat')
          IF(i.EQ.imax)THEN
              WRITE(3,102)time,C(i,jmax),q(i,jmax),v(i,jmax)
          ELSE
          END IF

      END DO
      CLOSE(2)
c      ...........Check Running Loop..............................

      IF(time.LT.300000) THEN
      GOTO 10
      ELSE
      GOTO 999
      END IF

C      .........Format for Input and Output Statements.......

101   FORMAT(F13.3,I3,I6,2E15.9,8E15.9)
102   FORMAT(F13.3,3E15.9,8E15.9)
999   STOP
      CLOSE(3)
      END
```

```fortran
c     ***********************************************************
      SUBROUTINE RK4(j,C,q,v,imax,jmax,KC,Kq,dt)

      IMPLICIT NONE
      DOUBLE PRECISION CC,qq,C,q,dC_dt,dq_dt,dt,KC,Kq,v
      INTEGER i,j,imax,jmax
      DIMENSION C(imax,jmax),q(imax,jmax),dC_dt(imax,jmax)
      DIMENSION dq_dt(imax,jmax),KC(4,imax,jmax),Kq(4,imax,jmax)
      DIMENSION CC(imax,jmax),qq(imax,jmax),v(imax,jmax)


c     ..............Define Parameter....................

      DO i=1,imax

         CC(i,j)=C(i,j)
         qq(i,j)=q(i,j)

      END DO

      Call ODEs_EQ(j,imax,jmax,CC,qq,dC_dt,dq_dt,v)

      DO i=2,imax

         KC(1,i,j)=dC_dt(i,j)
         Kq(1,i,j)=dq_dt(i,j)
         CC(i,j)=CC(i,j)+(dt/2)*KC(1,i,j)
         qq(i,j)=qq(i,j)+(dt/2)*Kq(1,i,j)

      END DO

         Call ODEs_EQ(j,imax,jmax,CC,qq,dC_dt,dq_dt,v)

      DO i=2,imax

         KC(2,i,j)=dC_dt(i,j)
         Kq(2,i,j)=dq_dt(i,j)

         CC(i,j)=CC(i,j)+(dt/2)*KC(2,i,j)
         qq(i,j)=qq(i,j)+(dt/2)*Kq(2,i,j)

      END DO

         Call ODEs_EQ(j,imax,jmax,CC,qq,dC_dt,dq_dt,v)

      DO i=2,imax

         KC(3,i,j)=dC_dt(i,j)
         Kq(3,i,j)=dq_dt(i,j)

         CC(i,j)=CC(i,j)+(dt)*KC(3,i,j)
         qq(i,j)=qq(i,j)+(dt)*Kq(3,i,j)

      END DO

         Call ODEs_EQ(j,imax,jmax,CC,qq,dC_dt,dq_dt,v)
```

```
      DO i=2,imax

         KC(4,i,j)=dC_dt(i,j)
         Kq(4,i,j)=dq_dt(i,j)

      END DO

      Return

      End
c     ********************************************************************
      SUBROUTINE ODEs_EQ(j,imax,jmax,c,q,dC_dt,dq_dt,v)

      IMPLICIT NONE
      DOUBLE PRECISION
c,q,dC_dt,dz,L,d2C_dz2,DL_A,v1,e_A,e_B,e_C,dq_dt
      DOUBLE PRECISION dC_dz,qstar,a1,b1,k1,db_A,db_B,db_C,DL_B,DL_C
      DOUBLE PRECISION b2,t1,d1,b3,t2,d2,RT,Pa,P,qqm,qm_B,Psat
      DOUBLE PRECISION v,dv_dz,Ct,qm_C
      INTEGER i,j,imax,jmax
      DIMENSION C(imax,jmax),q(imax,jmax),dC_dt(imax,jmax)
      DIMENSION d2C_dz2(imax,jmax),dC_dz(imax,jmax),qstar(imax,jmax)
      DIMENSION dq_dt(imax,jmax),v(imax,jmax),dv_dz(imax,jmax)
      DIMENSION qqm(imax,jmax),P(imax,jmax)


c        ..............Define Parameter.....................

      L =8.8                            ! Total bed length
      dz = L/(imax-2)                   ! Step size for length
      v1= 0.8920833                     ! Superficial velocity
      Ct=0.040902639                    ! P/RT
      RT=24.45388                       ! RT
      Pa=1.0133E+08                     ! atmospheric pressure


c        ....Parameter of Adsorber Zone-A (Activated Alumina)......

      db_A =0.73743          ! Bulk density of adsorbent Zone-A
      DL_A=0.028249704       ! Axial dispersion through Zone-A
      e_A =0.37              ! Void fraction of Zone-A

c        ...Parameter of Adsorber Zone-B (UI-94 1/8")...............

      db_B=0.66529           ! Bulk density of adsorbent Zone-B
      DL_B=0.027733066       ! Axial dispersion through Zone-B
      e_B =0.35              ! Void fraction of Zone-B

c        ......Parameter of Adsorber Zone-C (UI-94 1/16").........

      db_C =0.66529          ! Bulk density of adsorbent Zone-C
      DL_C=0.027170115       ! Axial dispersion through Zone-C
      e_C =0.34              ! Void fraction of Zone-C

c        ........Adsorption Isotherm Constant Parameter.........
      k1 =1.004023607E-04*0.5          ! Effective overall mass
                                         transfer coeficient
```

```
       j=j
c      ...................Zone-A (Alumina)......................

       a1=1.195                         ! For Freundlich-Isotherm
       b1=0.8313

       DO i=2,4

           qstar(i,j)=a1*(C(i,j)**b1)

           dq_dt(i,j)=k1*(qstar(i,j)-q(i,j))

           d2C_dz2(i,j)=(1/(dz**2))*(C(i+1,j)-2*C(i,j)+C(i-1,j))

           dC_dz(i,j)=(1/(2*dz))*(C(i+1,j)-C(i-1,j))

            dv_dz(i,j)=-((1-e_A)/(e_A*Ct))*dq_dt(i,j)*db_A/1.8

           v(i,j)=dz*(dv_dz(i,j))+v(i-1,j)

           dC_dt(i,j)=DL_A*d2C_dz2(i,j)-v(i,j)*dC_dz(i,j)
     1     -((1-e_A)/e_A)*dq_dt(i,j)*db_A/1.8-C(i,j)*dv_dz(i,j)

       END DO
c      .........Zone-B (Mol Siv 1/8")...............

       Psat=3169904.00
       b2=4.575E+09                     ! For A-D Toth Model
       t1=1.9707072
       d1=0.1042178
       qm_B=9.72                        ! monolayer capacity

       DO i=5,60

             P(i,j)=C(i,j)*RT*Pa

             qqm(i,j)=P(i,j)/(((b2+P(i,j)**t1)**(1/t1))
     1       *(1-P(i,j)/Psat)**d1)

           qstar(i,j)=qqm(i,j)*1.8*qm_B

            C(i,j)=P(i,j)/(RT*Pa)

           dq_dt(i,j)=k1*(qstar(i,j)-q(i,j))

           d2C_dz2(i,j)=(1/(dz**2))*(C(i+1,j)-2*C(i,j)+C(i-1,j))

           dC_dz(i,j)=(1/(2*dz))*(C(i+1,j)-C(i-1,j))

            dv_dz(i,j)=-((1-e_B)/(e_B*Ct))*dq_dt(i,j)*db_B/1.8

            v(5,j)=v(5,j)*e_A/e_B

            v(i,j)=dz*(dv_dz(i,j))+v(i-1,j)

           dC_dt(i,j)=DL_B*d2C_dz2(i,j)-v(i,j)*dC_dz(i,j)
     1       -((1-e_B)/e_B)*dq_dt(i,j)*db_B/1.8-C(i,j)*dv_dz(i,j)
```

```
        END DO
C       ..........Zone-C (Mol Siv 1/16").................

        b3=1.889E+14                                !For A-D Toth Model
        t2=2.8666471
        d2=0.144046
        Psat=3169904.00
        qm_C=9.83                                   ! monolayer capacity

        DO i=61,89

            P(i,j)=C(i,j)*RT*Pa

            qqm(i,j)=P(i,j)/(((b3+P(i,j)**t2)**(1/t2))
1          *(1-P(i,j)/Psat)**d2)

        qstar(i,j)=qqm(i,j)*1.8*qm_C

          C(i,j)=P(i,j)/(RT*Pa)

        dq_dt(i,j)=k1*(qstar(i,j)-q(i,j))

        d2C_dz2(i,j)=(1/(dz**2))*(C(i+1,j)-2*C(i,j)+C(i-1,j))

        dC_dz(i,j)=(1/(2*dz))*(C(i+1,j)-C(i-1,j))

          dv_dz(i,j)=-((1-e_C)/(e_C*Ct))*dq_dt(i,j)*db_C/1.8

        v(60,j)=v(60,j)*e_B/e_C

        v(i,j)=dz*(dv_dz(i,j))+v(i-1,j)

        dC_dt(i,j)=DL_C*d2C_dz2(i,j)-v(i,j)*dC_dz(i,j)
1          -((1-e_C)/e_C)*dq_dt(i,j)*db_C/1.8-C(i,j)*dv_dz(i,j)
        END DO

        i=imax
            qstar(imax,j)=qstar(imax-1,j)

            dq_dt(imax,j)=dq_dt(imax-1,j)

            d2C_dz2(imax,j)=0

            dC_dz(imax,j)=0

            dC_dt(imax,j)=dC_dt(imax-1,j)

            dv_dz(imax,j)=dv_dz(imax-1,j)

            v(imax,j)=v(imax-1,j)

        Return

        End
```

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