

Evaluation and performance measurement nano-pore fabrication and compare them with other devices and to determine important parameters especially surface for the different catalyst

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The specific surface measurements and obtain different surface properties such as pore size, total area, the specific surface area (total available area), pore size distribution, mean pore diameter, pore geometry and its Effect in nano-catalysis are a challenge is the catalyst industry's concerns. In this article the formulas Langmuir, BET and curves t , have been used to measure surface area and pore volume in the micro and meso structures by comparing and analyzing V_d (dead volume) measurement of its impact on different devices specific surface area. Statistical data analysis of the results was as follows: 1. Determine which contains inter-laboratory reproducibility and intra-laboratory measurement error of less than 0.004 is shown. Due to the uncertainty less than the critical value obtained from the tables, F , T essentially zero is acceptable Due to the accuracy and reproducibility of sample standard deviation and variance is acceptable range. To get the accuracy and consistency of this method compared with standard procedure, Article SRM was sent to four laboratories. The results were analyzed using analysis of variance with stat graphic software uncertainty device, instrument error and variance of the system was measured

Key words: pore size, catalyst, specific area, BET, Langmuir.

INTRODUCTION

A large variety of applications with modern technology are widely used in materials engineering for precise measurement of micro, meso and macro pores, these materials are used as catalyst to make paint, cement, carbon black, absorbent, absorbent in desiccant (absorbent material in air-handling), etc.[1]. This information includes porosity and overall surface area and powder surface area and the distribution of pores volume in different sizes.

Mainly errors occur in most of the techniques and methods of measurement that arise from three sources. The first source, due to the use of non-absorbent gas, because the chemical structure and physical properties, is used to calibrate the sample cell volume. Therefore, the absorbent gas is used after the calibrating by helium gas, and then, gas such as nitrogen, carbon dioxide or krypton is used along with the sample. In gas absorption, it is assumed that it should be absorbed by the sample. Also, gas absorbed by the cell walls is not considered. Therefore, cellular uptake is considered for measuring the absorption of sample. While it is possible that at the top surface of the sample, the increased error to be considered as the sample surface area and the amount of gas absorbed by the cell wall to be a greater fraction of the total gas adsorption.

The second source when the sample cell is immersed in a cooling device (for example, liquid nitrogen is often used to absorb and keep a constant temperature of the sample during the measurement process, therefore, the temperature leads to deviation from the ideal gas law.

Thus, the gas in the empty volume of the sample cell that becomes colder causes errors. Deviation from the ideal should be considered and corrected. However, due to the measurement of time, the bath should be carefully made, and because of that, the empty volume of the cell can't be measured accurately, the amount of correction for the volume of gas under non-ideal conditions is uncertain [1,2].

Trying to solve this problem in the past focused on minimizing, as much as possible, one of these cases is the advantage of reducing the amount of void volume in order to reduce measurement error, which this occurred with different degrees of the success in this thesis.

Major problems are related to speed, cost and difficulty to use a non-absorbent gas. The gasses which are better to use such as helium and krypton, are expensive, thereby the cost needed for measuring will be increased [3]. Moreover, apart absorption in the cell walls, determining cell volume at any time during calibration is time consuming. In addition, there is a method to prevent errors, and only using adsorbent gas, measurement problems can be solved to some extent [4].

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Accordingly, a device quite familiar with these problems is selected and solutions are provided for measuring empty volume, as well as the absorption of the sample cell walls and non-ideal gas behavior modification are considered using absorbent gas.

This study has attempted to fix errors and provide exact solution. Even now, effectively, with overcoming the above problems, and long-standing inherent problems to measure volume of pores, a simple, convenient and very effective solution is provided by which the accuracy of measurement has been increased, and the time consumed in performing measurements has been reduced. Use of gas for calibrating device and cell samples, and determining adsorption and desorption of properties of a powder sample. It is another object of this paper which related to gas absorbed by the sample cell walls to compensate

2. RESULT AND DISCUSSION

2.1. Calculations

The device includes a valve between manifold and sample cell to create liquid communications, and a pressure sensor is embedded to determine the pressure in the manifold and cell sample and a sensor for determining the temperature of manifold and the issues related to cell depletion. More accurate device includes a microprocessor having a memory. The volume of sample cell and sample cell walls absorption is stored in memory, so that, when an analysis of measurements is done on a sample in the sample cell, the sample cell volume and absorption of the sample cell walls can be done completely. A great advantage offered by this device is avoiding the need for a non-absorbent gas to calibrate the sample cell.

A device is provided to compensate for that portion of the sample cell where there is non-ideal absorbent gas. In an analysis of the absorption, offset method to measure the time-dependent error caused by the evaporation of liquid coolant, and changes caused by coolant level and increased temperature around the sample cell stem is very important. One method is the use of experimental results, and the use of time-dependent constant, including, changing the coolant level, and temperature changes of sample cells, change the cold area volume, and volume of gas absorbed to cell sample, and which should be used in the formulas [2].

$$N = N_C + N_W \quad (1)$$

$$\frac{\Delta P_m V_m}{R t(amb)} = \frac{\Delta P_C V_C}{R t_C} + \frac{\Delta P_W V_W}{R t_w} \quad (2)$$

N = moles of nitrogen gas from a manifold volume V_M in t temperature (ambient) is equal to mol transferred to the cold zone + mol transferred to cell heating zone in ideal conditions manifold pressure changes, when the gas is transferred to the cell. The changes in pressure inside the hot and cold zones of the cell samples are equal, although there is a temperature gradient between the hot and cold zones of the sample cell, the volume of gas in the sample cell can be provided, also the gas condenses has relationship with to the absorb density, the volume of gas condensate has been subtracted from the amount of cool zone gas volume.

$$N_a = \frac{\Delta P}{R} \left(\frac{V_w}{t_w} + \frac{V_c(1+aP)}{t_c} \right) \quad (3)$$

$$N_B = N_a - \frac{\Delta P}{R} \left(\frac{V_w}{t_w} + \frac{(V_c - M\rho^{-1})(1+aP)}{t_c} \right) \quad (4)$$

Subtracting the equation (6) from equation (7), and so on, the number of moles of gas that leave the manifold, the pressure to achieve the cell pressure at the time when there is no absorption, is given by:[3]

And to achieve the standard mode:

$$N_B = N_a - \frac{\Delta P}{R} \frac{(M\rho^{-1})(1+aP)}{t_c} \quad (5)$$

$$V^B = V^a - \Delta P M \rho^{-1} (1+aP) \frac{273.16}{77.4} \quad (6)$$

During an analysis of absorption, to determine the exact amount of absorbed gas, the pressure in the sample cell is increased, that is simply because of warming every part of the empty volume. However, during the measurement, the evaporation coolant and so, in cold zone, the volume is decreased, while, in the hot zone, it has increased. If the coolant level change is not compensated in some way, measuring the absorption of gas, which is a function of both pressure and time, will face with errors.

2.2 Measuring the volume of an ideal and dead gas

One of the factors used to calculate the volume of the dead gas, is calculating the volume of gas absorbed, the volume in the old system was calculated before or after the absorption system and this calculation has not been the same due to the changing situation, to change the volume depends on the nitrogen level changes over time, calculating the volume depends on two parts of container, the part that is in liquid nitrogen chamber, and the part

that is above the liquid surface to valve connected to manifold.

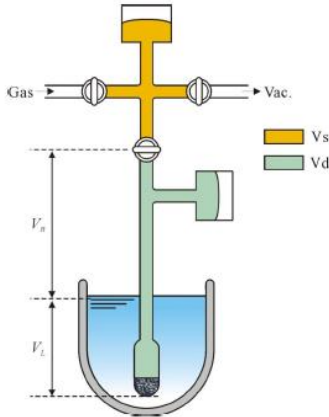


Figure 1. Schematic of BET device

Because, absorbent is nitrogen gas, because of volumetric absorption coefficient change with the upper part of the liquid surface to valve connected to the gas manifold, and according to the gas-liquid equilibrium equations governing situations, the temperature must be absolutely identical, to apply this assumption, three mathematical relations are shown as follows:

$$Vd = V_R + V_L \frac{T_R}{77.35} \quad (7)$$

$$Vd = V_R + V_L \frac{T_R}{77.35Z} \quad (8)$$

$$Vd - V_d = V_L \frac{T_R}{77.35} \left(\frac{1-Z}{Z} \right) \quad (9)$$

$$V_{dRT} = V_R + V_L \quad (10)$$

$$V_L = (V_d - V_{dRT}) \left(\frac{77.35}{T_R - 77.35} \right), Z = 1 + PB_{P=0.95} \quad (11)$$

On the other hand, when the sample cell is immersed in a coolant (for example, liquid nitrogen), often to absorb and maintain a constant temperature of the sample during the measurement process, so the temperature leads to deviation from the ideal gas law ($PV = ZNRT$). Therefore, the gas in the void volume of the sample cell which has become colder, causes the error.

Z is equal to one, when nitrogen gas is used and the temperature is 300K, and pressure is 100Pa. But in rotating, nitrogen temperature is 77 K and pressure is 100 Pa and Z is equal to 0.95, and in fact, it is 4% higher than the ideal value for the correction of the value measured in dead volume calculation as follows.

In this case, dead volume (Vd) changes as the surface level of liquid nitrogen changes. Normally, it is tried that the surface level of liquid nitrogen

does not change so that a constant value can be used for dead volume. There is no device for liquid nitrogen level control. Dewar vessel, which is used for sample cooling, is filled with liquid nitrogen (approx. 2 liter) before measurement. Liquid nitrogen evaporates and decreases gradually (up to 30 hours) during adsorption measurement. Due to the evaporation, dead volume of sample cell changes every second. In the dead volume change is measured as follows. A dead volume reference cell (an empty cell which is same as sample cell) is set to measurement port next to sample cell and immersed in liquid nitrogen (Fig.3). Dead volume of sample cell and dead volume reference cell are measured before adsorption measurement. In Fig.2, when the liquid nitrogen exists up to “level 1”, dead volume of sample cell is expressed by $Vd(smp)$, and dead volume and pressure of dead volume reference cell are expressed by $Vd(ref)(1)$ and $P(ref)(1)$ respectively. Also, when the liquid nitrogen has reduced down to “level 2” pressure of dead volume reference cell is expressed by $P(ref)(2)$. $Vd(ref)(2)$ (Dead volume of dead reference cell) and $\Delta Vd(ref)$ at level 2 are as follows:

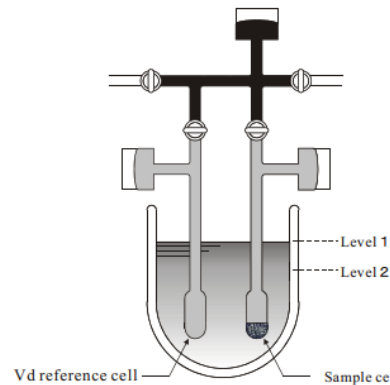


Figure 2. Schematic of sample device

$$V_{dref} = V_{dref(1)} \times \frac{P_{ref(1)}}{P_{ref(2)}} \quad (12)$$

$$\Delta V_{dref} = V_{dref} - V_{dref(1)} \quad (13)$$

$$\begin{aligned} V_{dsamp(2)} &= V_{dsamp(1)} + \Delta V_{dsample} \\ &= V_{dref(1)} + \Delta V_{dref} \end{aligned} \quad (14)$$

The pressure of adsorption gas that is dosed into the standard volume decrease as adsorption occurs. In volumetric method, adsorption amount is measured by calculating the difference between pressures before and after adsorption. $P_{i(n)}$ kPa is the pressure of adsorptive that is dosed from valve

A to Vs at nth measurement point. P1i(n) and Ws is the mass of adsorbent, and T means the absolute temperature of Vs. The gas amount (V2) that exists in Vd before adsorption can be calculated using P2e(n-1) (the equilibrium pressure inside Vd) as follows:

$$V_1 = \frac{(P1_i(1) - P1_i(1)) \times V_s \times 273.15}{101.30 \times W_s \times T} \quad (14)$$

Furthermore, V3, amount of the gas that remains in Vd at adsorption equilibrium is

$$V_2 = \frac{P2_e(n-1) \times V_d \times 273.15}{101.30 \times W_s \times T} \quad (15)$$

$$V_3 = \frac{P2_e(n) \times V_d \times 273.15}{101.30 \times W_s \times T} \quad (16)$$

The increased amount of adsorbed gas(ΔV) at nth measurement point can be expressed as follows

$$\Delta V = V_1 + V_2 - V_3 \quad (17)$$

Adsorbed amount at nth measurement point is the adsorbed amount at (n-1)th measurement point plus ΔV , thus it can be calculated with the following

$$V_n = V_{(n-1)} + \Delta V \quad (18)$$

2.3. Lifting System

Quantachrome Company [4] uses a lifting system and nitrogen container and bath lift in intelligent and automatic manner. Naturally, the surface should be considered as a constant value, but during measurement system, due to non-uniform changes and the lack of a gentle slope, the results are not considered identical (non-repeatability). The temperature is constant and does not change with a slope.

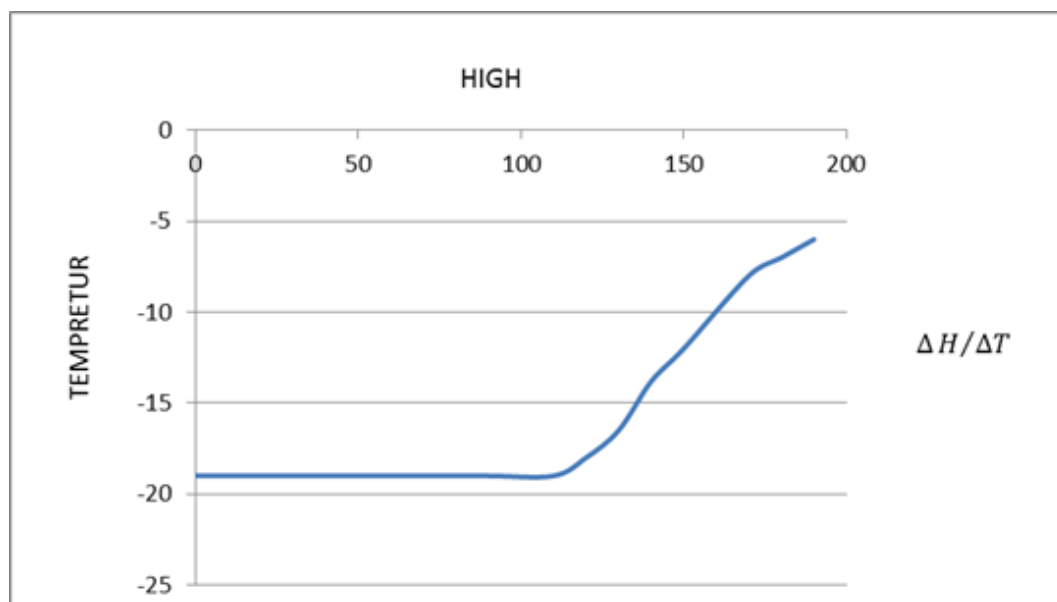


Figure 3. Changes in height (mm) compared to temperature ($^{\circ}C$)

2.4. Bell sorb control cell system

Vd was measured using a control container which at any moment measured temperature and all similar conditions, and according to the formulas derived in the previous section, the volume of gas adsorbed was calculated that has been extracted from U.S. patent method [5].

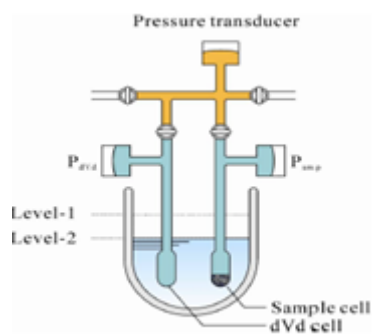


Figure 4. Mechanism of sample and control cell

3. ANALYSIS OF MEASUREMENT SYSTEMS

3.1. Determining repeatability

Repeatability studies in a laboratory only include operator and machine parts changes, so if a sample is tested by a device, the results were obtained using statistical software that its output is as follows:

Table 1. Results obtained using statistical software

	Hesgermad	Kimiazi
Count	10	10
Average	533.54	533.55
Standard deviation	0.0124722	0.0827312
Coeff. of variation	0.00233763%	0.0155058%
Minimum	533.53	533.36
Maximum	533.56	533.65
Range	0.03	0.29
Std. skewness	1.10903	-1.58264
Std. kurtosis	-0.588569	1.6668

Comparison of standard deviations

	Hesgermad	Kimiazi
Standard deviation	0.0124722	0.0827312
Variance	0.000155556	0.00684444
Df	9	9

Since the standard deviation is $S_m=0.0015$ and repeatability of the measuring tool is estimated about

$$\pm 3(0.0015) = \pm 0.0045$$

Thus, a device that is used for these sizes does not have an error greater than 0.0045 in any sizes.

3.2. Estimation of control charts for variables

Repeatability studies include measuring large numbers of samples by a device as well. To obtain separate estimates, more than two samples, each of

them should be repeated 10 times, in previous sections, this is done to obtain accuracy of device, however, if the standard deviation of read sizes and the average two equations are calculated, we can use Control chart R and data d (changes range) and statistical tables. Calculations of variance analysis for mean square for operators and machines show that the results are as follows:

These values indicate that the greatest changes in sizes are related to the devices, this is good, because in modern measuring devices, different

sources of measurement errors should be so small, so that should not be in conflict with the measured values significantly. A slightly different method is obtained using the analysis of two sizes obtained of each piece using graph R and dividing it by d, variance error estimation.

$$\sigma_{\alpha}^2 = \frac{s_m^2 + s_m^2}{2} - \frac{R^2}{d_2} \quad (19)$$

According to the graph R, after repeating the samples in a week and repeating the sample among other months, the average data was obtained equal to 0.023, for n = 7, upper and lower limit is obtained and the changes range of 20 samples were studied, and dynamic repeating pattern of out of control points was not observed, can be judged that the process is stable, only after ensuring the elimination of the causes of changes and process stability, which control charts can be used (base chart), and generally after drawing the sample mean, no signs based on the outside of the control of the graph was not observed, so measurement process is done under control.

$$M_s = \sigma^2 + n\sigma_y^2 + nk\sigma_b^2 \quad (20)$$

$$\sigma^2 = \frac{M_s \text{ opratpr} - M \text{ effect instrument}_s}{nk} \quad (21)$$

$$\frac{U_s}{S} = 2.95 \sqrt{\left(\frac{U_s}{S}\right)^2 + \left(\frac{U_v}{V}\right)^2 + \left(\frac{U_p}{P}\right)^2 + \left(\frac{U_t}{t}\right)^2 + \left(\frac{U_m}{m}\right)^2 + \left(\frac{U_{cal}}{v}\right)^2} \quad (23)$$

3.2.2. The effect of pressure

Because, the pressure gauge error is equal to 0.056, the item of pressure is not considered in

$$Vd(n) = \frac{(p1i(n) - P1e(2) \times V_s + P2e(n - 1) \times Vd(n - 1))}{P2e(n)} \quad (24)$$

The results of the effect will be least squares fitting straight line with a correlation coefficient of $r^2 = 0.997, 0.00548$.

$$\sigma_{\alpha}^2 = 0.0035 \quad \sigma^2 \beta = 0.00007. \\ \sigma^2 y = 0.001 \quad \sigma^2 \epsilon = 0.0005 \quad \beta \quad (22)$$

3.3. Uncertainty

Measurement uncertainty about quantitative feature of values distribution can be attributed to measurement issue. Uncertainty of measurement deals with defects in error analysis, and redefines how to interpret the measure completely. In this regard, it provides a quantitative index to put measured values in acceptable range, effective parameters on determining the effective level, and then the effects of each parameter correction should be determined, and sources of uncertainties should be identified.

3.2.1. Quantitative determination of the sources of uncertainty

The amount of uncertainty calculated using formulas, parts, volume, repetition, structure of temperature and pressure gauges and whatever was determined as a cause-effect relationship, was calculated as follows. Area factor is calculated in accordance with volume factor and formulas mentioned in the first section:

calculations, and this error should be entered in estimation of 0.05 of 95%, therefore the share of uncertainty is obtained according to the following calculations:

0.0112 = pressure uncertainty

Sample weight effect: By using the rectangular distribution rule, it is equal to:

3.2.3 Compound uncertainty calculation

The specific surface area was calculated by using all effective parameters on the relationship between cause and effect in accordance with the following formula (Number before radical is considered as a coverage factor, which is explained in section two):

Table 2. Results of uncertainty

	Surface adsorption	m3/g uncertainly	12% surface
Sample 1	533.50	9.587	495.7
Sample2	10.22	9.06	9.06

As can be seen, the values obtained should be calculated according to standard, it is acceptable for all measurement levels.

The results of the four labs with different devices on different devices are presented in the table below, carbon black standard with the amount

bet surface area 528.34 was purchased from Merck and sent to four laboratories to evaluate using the statgraphics software and calibrating a measurement device is the most reliable method for measuring that device. [102]

Table3. AOVA Results of four laboratories

	Bel sorb	Fintac china	Hms sorb	qntocrum	crm
b1	533.54	533.65	533.54	533.61	533.54
b2	533.54	533.53	533.56	533.65	533.54
b3	533.59	533.57	533.56	533.61	533.54
b4	533.59	533.57	533.53	533.59	533.54
b5	533.59	533.52	533.53	533.59	533.54
b6	533.59	533.61	533.54	533.59	533.54
b7	533.52	533.51	533.53	533.59	533.54
b8	533.52	533.64	533.53	533.53	533.54
b9	533.52	533.36	533.53	533.59	533.54
b10	533.55	533.54	533.55	533.55	533.54

As it can be seen, the variance ratio obtained from experimental results (2.34) is smaller than the table or critical values (2.57). The null hypothesis should be accepted, it means that, the results should be trusted, and so it can't be said with certainty that changes between categories have a significant impact on the yield at a confidence level of probability 0.05, and the variance between categories and laboratory is clearly 0.0018 less than the critical value. So inter-laboratory variations have a significant impact on the efficiency of the probability $p = 0.05$.

4. ACCURACY

Due to standard material, we can achieve absolute error and relative error. However, if the standard material is not available, we can use the En parameter, if obtained parameter is less than one, whatever is less, it will have good accuracy, and if it is equal to or greater than one, this doesn't

have good accuracy, En parameter can be obtained as follows:

$$E_N = \frac{\overline{X}_{SAMPLE} - \mu_{REF}}{U_{SAMPLE}^2 - U_{REF}^2} \quad (25)$$

Because En amounts are equal to 0.18, and less than one, this method was accurate, and confirmed. However, the calibration only evaluates the measurement error of a device, and repeatability errors are usually very time-consuming and costly. Another solution is to use a relatively simple method for two or more measurement devices, in order to compare the two devices, the number of n and for greater certainty and four devices from different companies were measured. The difference in accuracy values of two devices can be done by typical two-sample t-test. The following instruction shows the stages of comparison test of the variance for paired data, (1) calculate the correlation

coefficient between the standard deviation and changes range d. Perform a regular test on r item.

$$r = \frac{S_i D_i - \frac{\sum d S_i d_i}{n}}{\sqrt{s^2 i - \frac{\sum s^2}{n}} \times \sqrt{\sum d i - \frac{1}{n d^2}}} \quad (26)$$

In order to compare mean (paired data) as = t and standard deviation, the difference between changes for existing device to compare the accuracy of two measurement devices, n = 5 correlation between r for standards test was read. The difference at importance level of the critical values of freedom degree according to the table is 2.060 but the absolute value of t = 8 is greater than the critical value, we conclude that the accuracy (has been measured by the standard deviations) of two measurement devices are different.

$$S = 0.07 \quad d = 0.0014 \quad r = 0.8879$$

$$t = \frac{-0.8\sqrt{8}}{\sqrt{1-0.878}} = -8.797$$

$$\frac{d\sqrt{n}}{s_d} = \frac{0.0148}{0.0704}$$

But, according to possible differences test in accuracy of sizes and for important level and confidence of 0.05 with t=2.06, since the amount calculated, 1.05 is less than the table, there is no significant differences between the data read by two devices.

4.1. Determining bias

Analysis method bias, while within the enterprise and by using a standard sample is examined, so the analysis of variance and determining bias between laboratories can be measured:

Table4. Results of comparison between two laboratories by t test
t-Test: Two-sample assuming unequal variances

	Hesgermad
Mean	533.54
Variance	0.000155556
Observations	10
sd	0.012
U(REC)=Sd/√10	0.03
t Stat= $\frac{1-RCE}{UREC}$	3.3
P(T<=t) one-tail	0.094584641
t Critical one-tail	1.782287556
P(T<=t) two-tail	0.189169283
t Critical two-tail	2.17881283
R%	90%

As we see, for a two-ways test with degrees of freedom n-1, the value obtained is greater than from experimental results, 3.3, with the amount taken from table and the critical value of 2.06, then we can say with certainty that there is considerable difference between the two ways, and the null hypothesis is not acceptable and there are significant differences between two methods for measuring surface area.

5. EVALUATION OF SOFTWARE

Standard curves obtained from the device prove the following:

This reality has not quite match the experimental and theoretical results, and is consistent with IUPAC standard curve on the Isomer curves in terms of compliance, for example, here, the carbon black curve is 1-type isotherm, adsorption and desorption isotherm curves of SiO₂ is 4-type IUPAC, but is consistent with data obtained from

Merck certificate. According to standard curves for standard carbon black material, medium holes can be guessed, such as, holes with loose openings and due to the lack of waste disposal condensate liquid are easily removed, and pores diameter cavities are mid-size, it should be predicted that condensed cavity is controlled by hemispherical cylinder, as soon as the cavity is filled, while, evaporating liquid occurs from a hemispherical arc, which gives the different values according to the Kelvin equation for the condensation and evaporation. Based on the BJH, HK and SF theories and analysis of results, the samples with a frequency of approximately two nanometers for the carbon black samples and for SiO₂ prove that it is consistent with MP theories.

According to hysteresis charts, the sample has Type H4 hysteresis for SiO₂ which indicates a mixture of meso and micro holes and is non-uniform. According to the isotherm curves, due to

partial pressure change, when the number begins from 0.5 at the beginning of hysteresis, we can find that there is meso-hole or holes Average distribution, and for SiO_2 when the beginning of opening above 0.6 is closed, the openings of these pores is widened, liquid exits the cavity, faster, and thus, hysteresis closed curve is too small, also it will be more for micro-holes.

According to the hysteresis curves, and their shapes type, the curves obtained by the device is presented in accordance with to theories, and comply with the IUPAC standard curves with D shape which is a mixture of type A and B Type of inkjet tank holes.

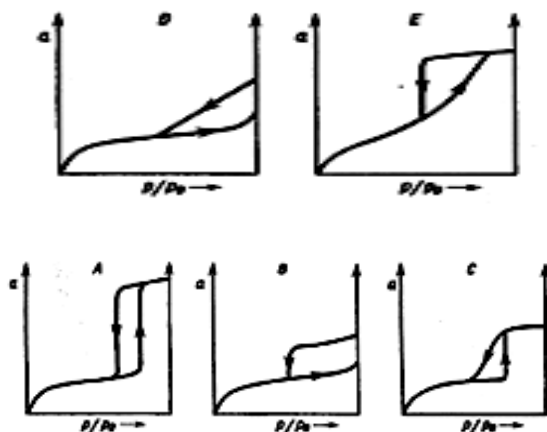


Figure 5. Kind of hysteresis curve for different catalysts.

As seen in data at the bottom of the charts, below the excell charts, all charts are available and standards, including in terms of user convenience are observed, and are written for not having software computational errors, including various kinds, a program to communicate with computer hardware and convert the pressure data to adsorbed volume, and the second program is responsible for processing data and drawing graphs. Given that, a long time is needed to obtain data (over eight hours), we can take necessary measures to previous data processing by the second program, this is observed for all devices manufactured, also the data processing Excell software is accessible and these charts can be drawn easily.

5.1. Measuring the comparative samples

To assess the different holes openings, four types of commercial activated carbon are tested, as can be seen, all these examples have Micro-cavity pores. All samples follow the type-1 isotherm, and attract potential impact on the chemical potential at the point where there is not condensed cavity, can be clearly seen. Also, the impact of the curves on the

thickness of the adsorbed film, and there is not the hole condensed, so absorption of film laminated on a porous solid surface is similar. Samples are fundamental differences in terms of surface area, but in terms of the pore diameter of all samples is about 1.3 nm, and the parallel plates or cylindrical pores can be considered. It is clear that the frequency of meso holes for sample Sa is equal to 356, for sample S7 is equal to 72, for mc is equal to 652, for SKH is equal to 687, which looking at the curves, it can be predicted that the overall surface Sa is more than the others. In fact, it can be said that, the c value is greater than the other samples, and tends towards the infinite. Sample sa7 of 4H – type cycles has thinner pores, and often pores are observed within the micro, as well as in particle size BJH is clear that which it is 1.3 nm (micro-pore), and in this size of the cavity, especially at clogging low pressure, there is not blocking the holes. In sample Sa, compared to sample sa7, it has bigger pores and even more frequently.

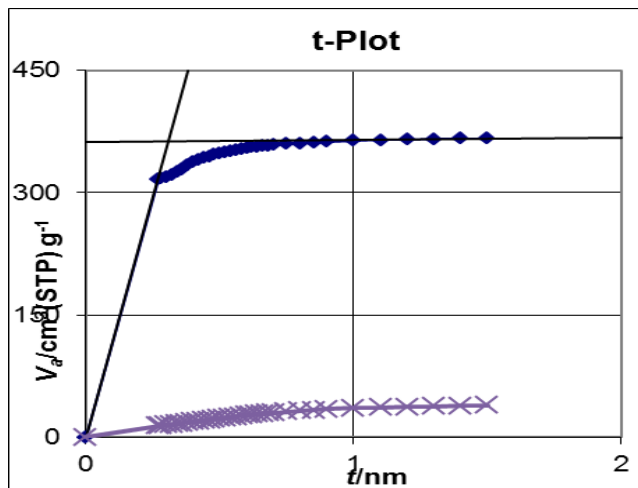


Figure 5. Camper curve t between different carbon black

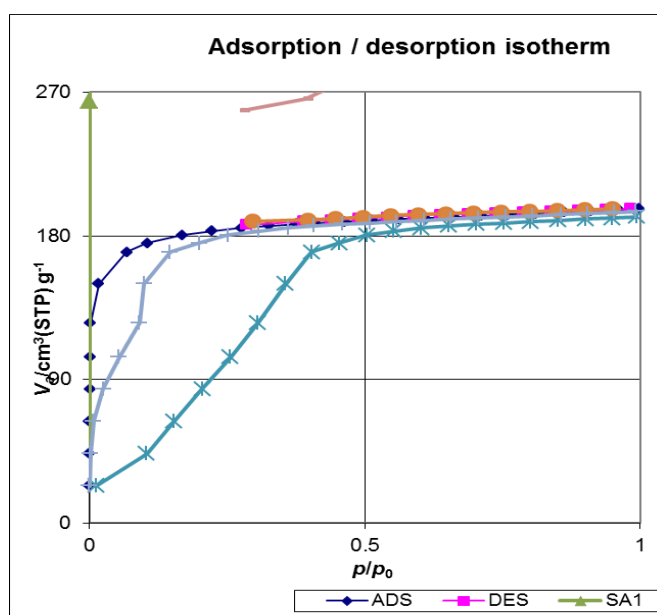


Figure 6. Camper curve between different carbon black

This can be guessed in charts t in such a way that in the sample Sa shows BET volume, surface of holes larger than the sample mcm, skh and the overall perimeter of the area which has strong relationship with the slope of the curve and tiny

pores is obtained from difference between the outer surface perimeter and the overall perimeter of BET. Now, we look at the results of isotherm curves:

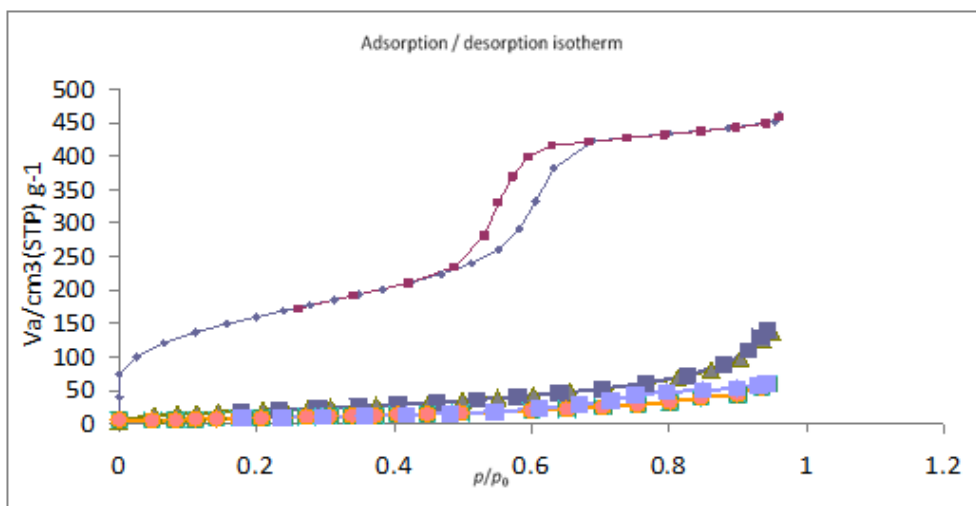


Figure 7. BET diagram.

CONCLUSION

In the previous examples, almost a sample was evaluated, and for checking conformity with the IUPAC six series samples, various samples were measured. What is certain is that the phenomenon of hysteresis occur for holes with open ends, and for charter holes (cylindrical-cone), the hysteresis phenomenon will appear to be quite rectangular, this phenomenon, in cases where holes are in form of a closed cylindrical charter, as a drop of a source of tension is obtained from a vacuum system, and it occurs on the samples at the beginning of hysteresis loop under a partial pressure of 0.5, and indicates the micro sample, and if is above 0.6, indicates the presence of meso-sized pores, which experimental results confirm the existence of such holes, for curves with no hysteresis loop, and the lack of open pores or pyramid-like pores, such an issue is true for lower two samples, because of thermodynamic conditions holes, it has low distribution, as if we refer to the results of the BET, it confirms that the number is less than 100.

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