

# The Potential Application of Modified Spruce-based Activated Carbon for Adsorption Chillers

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## Abstract

In effort to contribute to the ongoing research of green alternative cooling solutions, this research work studied the adsorption performance of spruce based activated carbon - WA TONG on methanol and the potentiality of this adsorbent pair to be adopted and applied in adsorption chillers. With focus on the low thermal energy powered chillers, the analysis was carried out in sub atmospheric conditions at four adsorption temperature points of 30, 40, 50 and 60 °C. Under the same conditions, WA TONG sample performance was tested against the two commercially available samples CWZ22 and CWZ22P and also a comparative evaluation with the high performing activated carbon methanol pairs available in literature was made. Methanol adsorption uptake for WA TONG sample was found to be as high as 60 % of adsorbent weight at adsorption temperature of 30 °C and relative pressure of 1.

*Keywords: activated carbon, adsorbents, adsorption chillers, cooling*

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## Potencial aplicação de carvão ativado modificado produzido a partir de abeto para chillers de adsorção

### Resumo

Com o intuito de contribuir para a investigação de soluções alternativas de arrefecimento, este trabalho estuda o desempenho de adsorção do carvão ativado produzido a partir de abeto - WA TONG em metanol e o potencial deste par adsorvente ser adotado e aplicado em chillers por adsorção. Com foco nos chillers de baixa energia térmica, a análise foi realizada em condições subatmosféricas em quatro níveis de temperatura de adsorção de 30, 40, 50 e 60 °C. Sob as mesmas condições, o desempenho da amostra WA TONG foi testado em comparação com as duas amostras comercialmente disponíveis CWZ22 e CWZ22P e também foi feita uma avaliação comparativa com os pares de metanol de carvão ativado de alto desempenho disponíveis na literatura. A absorção de adsorção de metanol para a amostra WA TONG foi de 60 % do peso de adsorvente à temperatura de adsorção de 30 °C e pressão relativa de 1.

*Palavras-chave: carvão ativado, adsorventes, chillers de adsorção, arrefecimento*

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## 1. Introduction

In recent years, most parts of the world have been experiencing very hot summers and some of the harshest heat waves to date. The annual global temperature keeps rising. Since 1981, the global annual temperature has been increasing at an average rate of  $+0.18\text{ }^{\circ}\text{C}$  per decade [1]. In the light of climate change threat and ever-increasing unpredictability of weather conditions across the globe, humans are looking for ways to mitigate the effects of this new reality on their well-being and overall economic productivity. The prominent problem people are facing is heat discomfort due to high temperatures. On a local scale, since their inception, the electric air-cooling systems have been the go-to solution in dealing with this challenge. However, the poor efficiency of these systems leads to high power consumption which in the grand-scheme of energy cycle happens to be counterproductive towards the mission of reducing global emissions. As of 2018, the use of air conditioners and electric cooling appliances accounted for 20% of the total electricity consumption in buildings worldwide [2]. As the world's emerging economies mainly in tropical hot areas, keeps growing along with their demographic growth, the global cooling demand is expected to rise. As income and standards of living improve, people will naturally want to ensure thermal comfort in their homes regardless of the ambient state. This will impose a significant strain on local electricity grids as the overall energy demand increases and it will in turn drive up the conventional firing emissions as long as the renewable alternatives aren't self-sufficient yet.

As of 2019, conventional cooling installations and appliances accounted for about 10% of all global greenhouse gas emissions which at the time was more than twice the emissions from aviation and maritime vessels combined [3]. Most widely available cooling solutions on the market are predominantly of vapour compression refrigeration system (VCRS) type which rely heavily on use of refrigerants like chlorofluorocarbons (CFCs), hydrofluorocarbons (HFC) and hydrochlorofluorocarbons (HCFCs) which are one of the main agents for ozone layer depletion [4]. Due to in time leakage that exists within these

VCRS, the refrigerants come into contact with the atmosphere and reacts with ozone layer which results into ozone depletion. Leakage leads to an unwanted refrigerants production cycle to compensate for lost amounts of refrigerants, on estimate only 35% of total refrigerants produced is for the new air conditioners and refrigerants, the rest 65% comes for refill purposes [4].

One promising alternative cooling solution is the adsorption-based cooling systems namely adsorption chillers. Currently, adsorption cooling solutions are still in the research phase and most proposed prototypes are at a viability market competitiveness disadvantage in comparison to alternative cooling solutions due to technical performance limitations of adsorption chillers. The core component of an adsorption system is the adsorbent adsorbate pair applied in the system and this is the utmost defining factor on the system performance. For the system to be practically viable, adsorbent must have high enough sorption performance, good thermal-physical properties, be abundantly available and cheap. Hence, the objective of this thesis was to explore whether a biomass spruce based activated carbon can be the adsorbent to fulfil the above highlighted premise and also to see how such adsorbent fair in comparison to the best activated carbon samples currently available on the market.

This work highlights the air conditioning challenge and the potential impact such niche of energy consumption poses to the environment. Three activated samples were considered this study and the comparative analysis of their methanol adsorption performance was made. The preparation method and treatments of the studied adsorbent samples are explained. The description of preparation procedure of the activated carbon sample WA TONG, from the precursor material spruce lignin to the potassium hydroxide (KOH) activation of the hydrochar was outlined. Density, porosity and XRD characterization of the samples were done. The experimental setup of the sorption performance analyzer is also explained. And lastly, the adsorption performances results are presented.

## 2. Materials and experimental methods

### 2.1. Preparation of activated carbon sample

The two of the samples CWZ22 and CWZ22P are commercially available activated carbon samples obtained from "CARBON" branch of the company ELBAR – KATOWICE Sp. z o. o. These are produced based on charcoal precursor materials and are activated by steam. The third sample is WA TONG activated carbon produced from lignin. The WA TONG sample was experimentally produced at AGH by Dr. Sztekler and his colleagues from spruce derived lignin precursor material and was activated chemically using potassium hydroxide (KOH). KOH activation approach aims to produce activated carbon with a narrow pore size distribution and the development of effective porosity. It is believed that the activation mechanism with alkali metals such as KOH relies on the fact that alkali metals act as an input catalyst in the carbon network, an electron donor, during the gasification reaction [5]. The preparation method and activation procedure for this sample are described below:

i. Kraft lignin produced from spruce by the CleanFlow Black Lignin (CFBL) process was used as the carbon precursor material [6]. Before the experiment, the lignin was first dried at room temperature to a moisture content level of about ~2%. Then the dried lignin was grinded and sieved to the size of particle diameter  $d < 0.125$  mm.

ii. 10 g of kraft lignin was then mechanistically mixed with 1.9 g FeSO<sub>4</sub>.

iii. The resulting mixture then underwent carbonization at 550 °C for 1 hour.

iv. Followed by mixing of Fe-hydrochar product with KOH (1:3 ratio) by way of mechanical stirring overnight at room temperature.

v. Then the mixture was dried at 105 °C.

vi. Followed by activation at 800° C for a duration of 3 hours.

vii. The resulting product was then washed using distilled water until pH reach to neutral levels and then drying of the product at 95 °C in an oven for 24 hours duration.

### 2.2. Characterization of activated carbon sample

**Porosity and density analysis:** As a porous material, activated carbon samples have both apparent density and true density. Both properties were measured for all three studied samples. Analytical technique of gas displacement pycnometry (AccuPyc II helium pycnometer) was used to determine the true density of the samples. True density was determined based on the skeletal volume of the sample measured through helium displacement penetrating into the open pores in the studied lump sample. By the method of pycnometer quasi-liquid displacement apparent density was determined. A GeoPyc 1360 quasi-liquid pycnometer was used for this. The test consisted in determining the volume of a lump sample in dry conditions. The estimated true and apparent density for WA TONG sample were 2.104 g/cm<sup>3</sup> and 0.8272 g/cm<sup>3</sup> respectively. The WA TONG sample porosity ( $\epsilon$ ) and pore volume of about 60.68% and 0.73 cm<sup>3</sup>/g, respectively was calculated from the obtained values of sample apparent ( $\rho_b$ ) and true( $\rho_p$ ) density through the equations below:

$$\epsilon = (1 - \rho_b/\rho_p) \times 100 \quad \text{equation 1}$$

$$\text{Pore volume} = \frac{\epsilon}{100} \times \frac{1}{\rho_b} \quad \text{equation 2}$$

**X-Ray diffraction phase analysis:** The phase composition and crystal structure analysis of the considered powdered activated carbon samples were done at room temperature using the powder X-ray diffraction method (XRD) with CuK $\alpha$  radiation. Panalytical Empyrean diffractometer equipped with PIXcel3D detector was the device used, with the  $2\theta$  angular range of 10-110 degrees, a  $2\theta$  step size of 0.013 degrees and a scan step time of 97 seconds. The obtained XRD monographs for the three samples WA TONG, CWZ22 and CWZ22P are presented below:

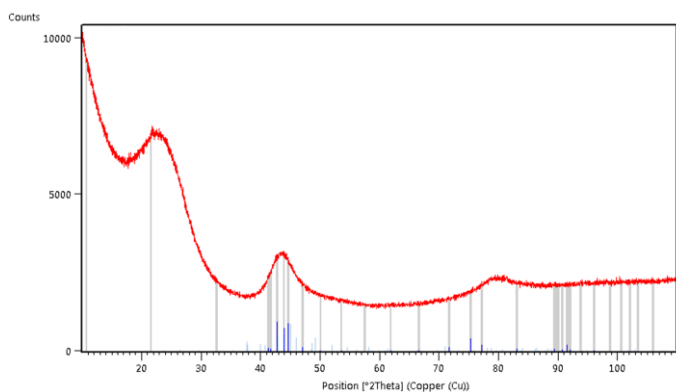


Figure 1 – XRD pattern of CWZ22 sample

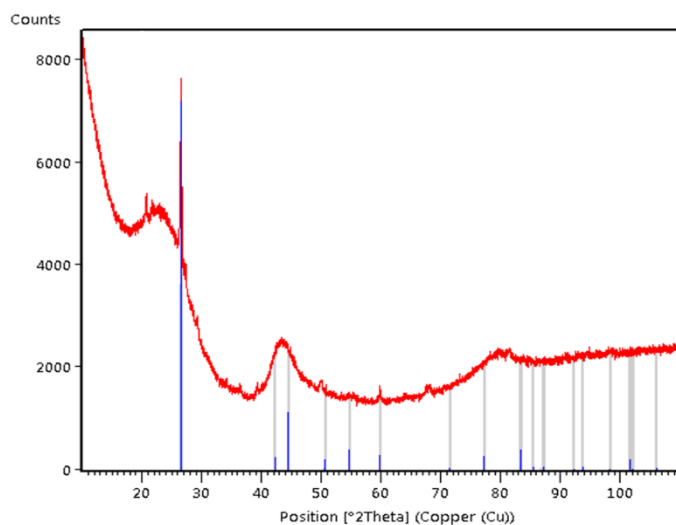


Figure 2 – XRD pattern of CWZ22P sample

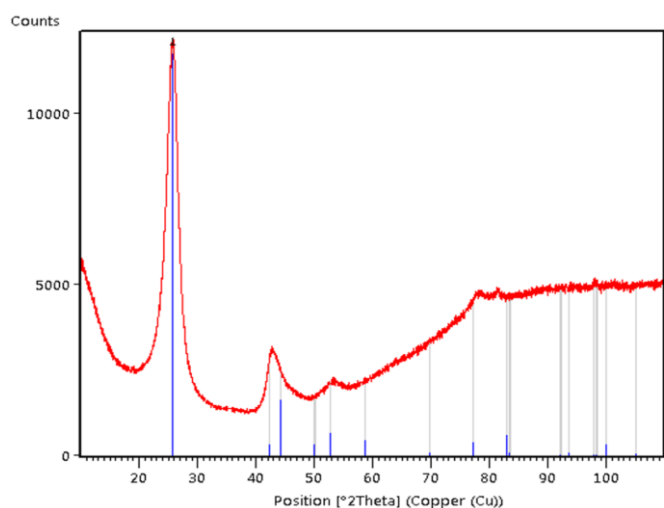


Figure 3 – XRD pattern of WA TONG sample

The presence of diffraction sharp peaks on the above XRD graphs is indicative of the existence of a well aligned layer of crystalline carbonaceous structure in all three studied samples [7]. On all obtained monographs the noticeable strong and weak diffractions peaks appear around 2Theta ( $2\theta$ ) degree positions of 25 and 45 respectively. WA TONG

sample exhibits sharper diffraction peaks than the commercial samples (CWZ22 and CWZ22P), this indicate that WA TONG sample has bigger crystallite size [8]. The almost nonexistence of many sharp peaks across obtained monographs informs that all three samples are predominantly of amorphous structure which aligns with the fraction predominance of carbon in activated carbon [7]. The lower crystallinity level in the sample, the more amorphous structure of the samples and consequently the higher probability of having larger specific surface area which is a preferred feature for adsorption application [8].

### Setup for adsorption performance analyzer

The sorption performance of samples was studied using the novel Dynamic Vapor Sorption (DVS) vacuum analyzer. It is based on the principle of gravimetric analysis of the sample whereby the sorption properties of a sample like activated carbon are measured in a preset condition (pressure and initial temperatures) as a controlled flow of a given adsorbate passes through the sample accompanied with monitoring intime weight change of the sample.



Figure 4 – DVS Vacuum gravimetric sorption analyzer [14]

In this experiment, the process goes as follows: first before opening the DVS chamber to set up the samples the system pressure was levelized to room atmospheric pressure using the pump control valve at the rate of 0.1 torr/s up to 10 torr (10 mmHg) and then at rate of 1 torr/s till to the room atmospheric pressure of about 750 torr was reached. Then the activated carbon sample mass of about 20 mg was measured and placed on the sample slide paired with an empty control reference pan inside the chamber. The vapor flask was filled with methanol as it is the adsorbate choice

for the study. After the chamber is sealed tight and the pump atmospheric valve is closed. Then the rotary pump is turned on to create vacuum in the system.

The sorption analysis for each sample were carried out on four different adsorption temperature points (30, 40, 50 and 60 °C) depending on which the saturation vapor pressure for methanol were set in the predefined DVS software method with a set of parameters like system temperature, process time duration together with step increment, and adsorbate vapor flow rate. In our case these parameters included preheating and cooling for 60 minutes each and sorption timestep duration (20 minutes per increment). After setting up the sample and making sure the vacuum state is assured in the system. The process starts with drying and in-situ degassing of the sample (preheating), followed by an about hour cooling of the sample to the predefined temperature point, and then the adsorption process starts with adsorption rate varying incrementally with the relative pressure change in the system till the adsorbate saturation pressure is reached at P/Po of 100% the point at which maximum desorption rate and minimum adsorption rate are observed. The analysis results were obtained in form of adsorption isotherms and adsorption kinetics curves.

### 3. Results and Discussion

Adsorption performance of the three activated samples on methanol were put to test under vacuum condition using the DVS apparatus as mentioned above. The experiment was carried out at four different temperature points (30, 40, 50, and 60 °C) for each sample and a DVS method was predefined in accordance with the methanol saturation pressure (Po) at each of the four points. The analysis results were illustrated in form of adsorption isotherms and kinetics curves. Of all the four points of analysis highest adsorption uptake were observed at a 30 °C adsorption temperature.

#### 3.1. Spruce-based sample – WA TONG

At adsorption temperature 30 °C, reference sample weight - 21.047 mg, methanol saturation pressure at 30 °C - 21.83 kPa, below are presented the isotherm and kinetics curve results.

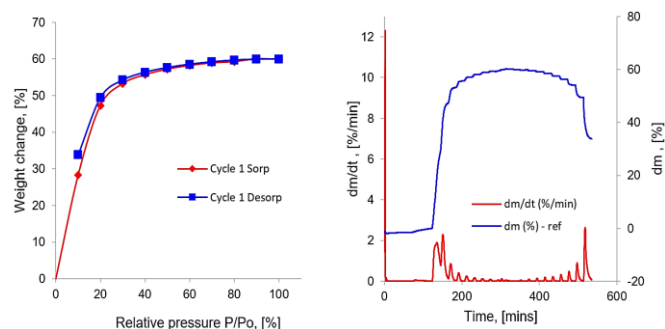


Figure 5 – Methanol adsorption on WA TONG at 30 °C. a) isotherm, b) kinetics

At adsorption temperature 39.7 °C, reference sample weight – 5.039 mg, methanol saturation pressure at 40 °C - 35.38 kPa.

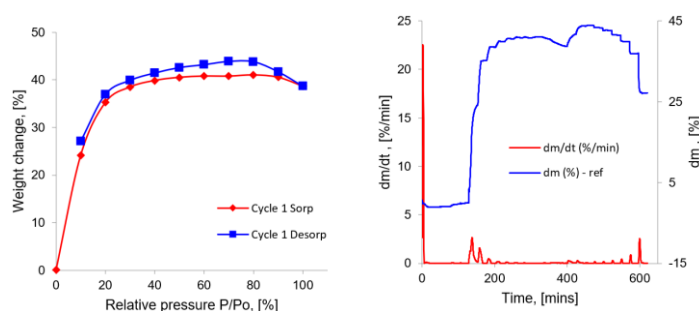


Figure 6 – Methanol adsorption on WA TONG at 40 °C. a) isotherm, b) kinetics

At adsorption temperature 49.3 °C, reference sample weight – 5.25 mg, methanol saturation pressure at 50 °C – 55.47 kPa.

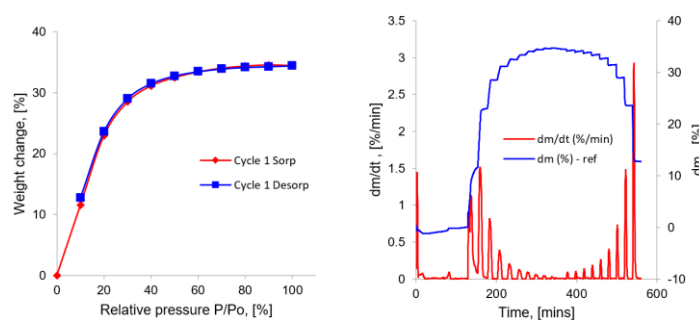


Figure 7 – Methanol adsorption on WA TONG at 50 °C. a) isotherm, b) kinetics

At adsorption temperature 58.8 °C, reference sample weight – 5.49 mg, methanol saturation pressure at 60 °C – 84.40 kPa.

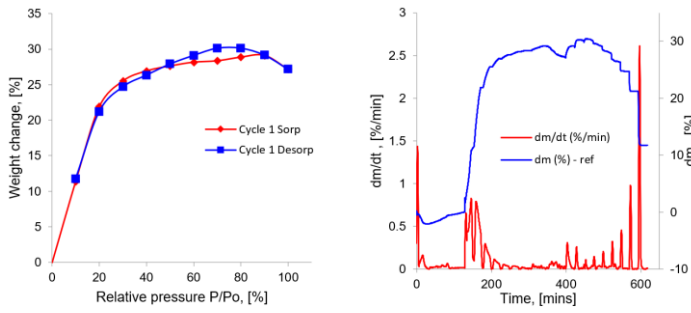


Figure 8 – Methanol adsorption on WA TONG at 60 °C. a) isotherm, b) kinetics

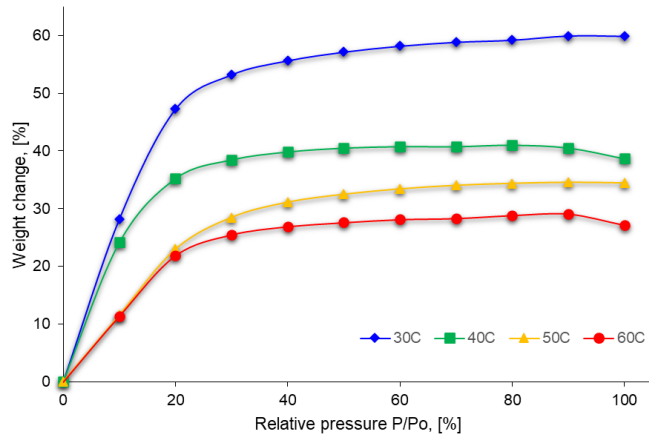


Figure 9 – An illustrative stack depiction of WA TONG sample adsorption isotherms at different temperatures

For sample WA TONG, the consistent trend from the above isotherm plots is that the sample exhibits isotherm of type I. its methanol adsorption uptake decreases with an increase of adsorption temperature. Hence the maximum uptake of about 60% adsorbent weight was on a 30 °C isotherm curve at saturation pressure condition. On isotherms at 40 and 60 °C, there's a deviation from the expected rising or plateauing isotherm curve trend around the saturation pressure zone. This led to what seems like a hysteresis between adsorption and desorption curves for which the plausible explanation is the insufficient equilibrium time. The sorption rate curve patterns on the respective kinetics matches the spotted deviations. Looking at the kinetics chart one can infer that with adjustment of adsorption step time (a bit larger timestep at the beginning of adsorption), the observed loop could have been mitigated and which would consequently lead higher adsorption uptake at even lower relative pressure.

### 3.2. Commercial samples – CWZ22P and CWZ22

**CWZ22P sample** – at adsorption temperature 30 °C, reference sample weight – 22.29 mg, methanol saturation

pressure at 30 °C - 21.83 kPa. Below are presented the isotherm and kinetics curve results.

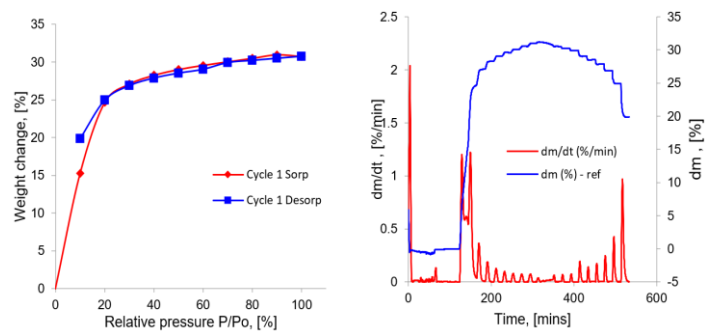


Figure 10 – Methanol adsorption on CWZ22P at 30 °C. a) isotherm, b) kinetics

At adsorption temperature 39.7 °C, reference sample weight – 22.34 mg, methanol saturation pressure at 40 °C - 35.38 kPa.

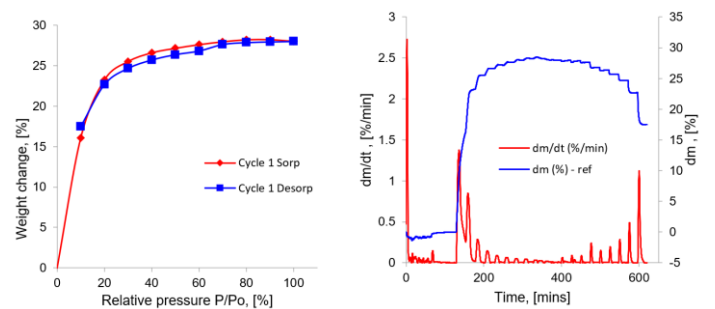


Figure 11 – Methanol adsorption on CWZ22P at 40 °C. a) isotherm, b) kinetics

At adsorption temperature 49.4 °C, reference sample weight – 22.29 mg, methanol saturation pressure at 50 °C – 55.47 kPa.

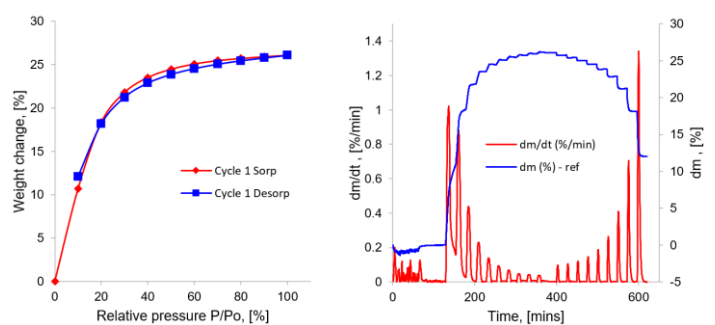


Figure 12 – Methanol adsorption on CWZ22P at 50 °C. a) isotherm, b) kinetics

At adsorption temperature 58.6 °C, reference sample weight – 22.25 mg, methanol saturation pressure at 60 °C – 84.40 kPa.

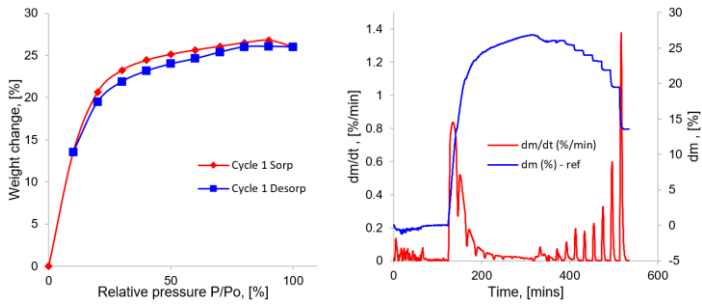


Figure 13 – Methanol adsorption on CWZ22P at 60 °C. a) isotherm, b) kinetics

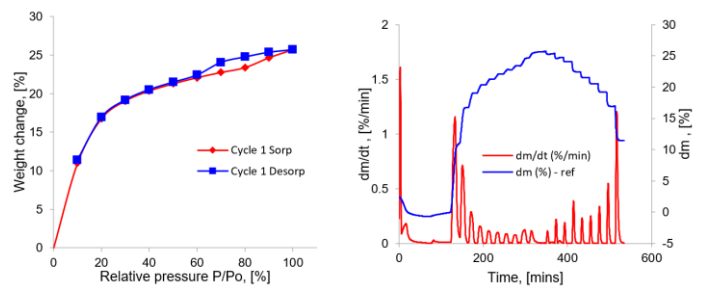


Figure 16 – Methanol adsorption on CWZ22 at 40 °C. a) isotherm, b) kinetics

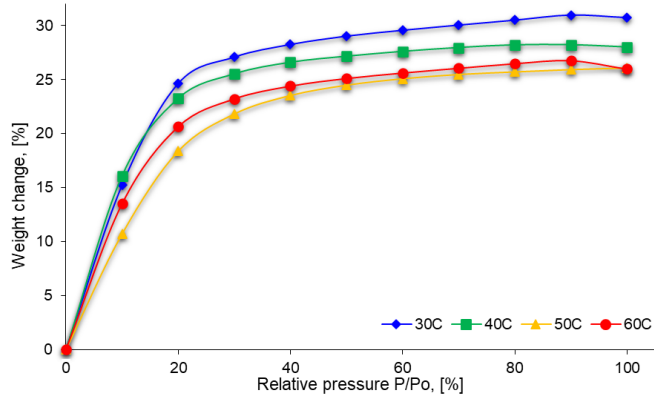


Figure 14 – An illustrative stack depiction of CWZ22P sample adsorption isotherms at different temperatures

**CWZ22 sample** – At adsorption temperature 30 °C, reference sample weight – 18.76 mg, methanol saturation pressure at 30 °C – 21.83 kPa.

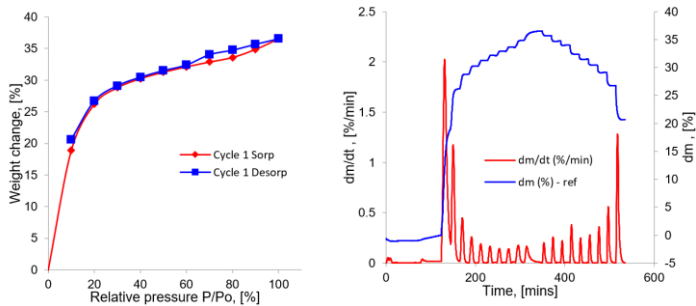


Figure 15 – Methanol adsorption on CWZ22 at 30 °C. a) isotherm, b) kinetics

At adsorption temperature 40 °C, reference sample weight – 19.17 mg, methanol saturation pressure at 40 °C - 35.38 kPa.

At adsorption temperature 49.6 °C, reference sample weight – 18.73 mg, methanol saturation pressure at 50 °C – 55.47 kPa.

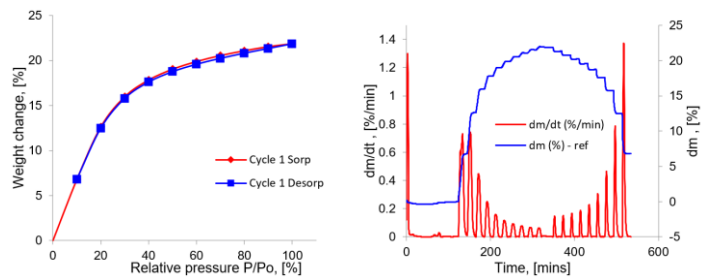


Figure 17 – Methanol adsorption on CWZ22 at 50 °C. a) isotherm, b) kinetics

At adsorption temperature 58.6 °C, reference sample weight – 20.41 mg, methanol saturation pressure at 60 °C – 84.40 kPa.

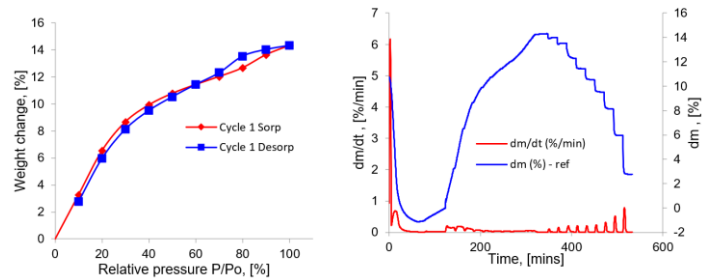


Figure 18 – Methanol adsorption on CWZ22 at 60 °C. a) isotherm, b) kinetics

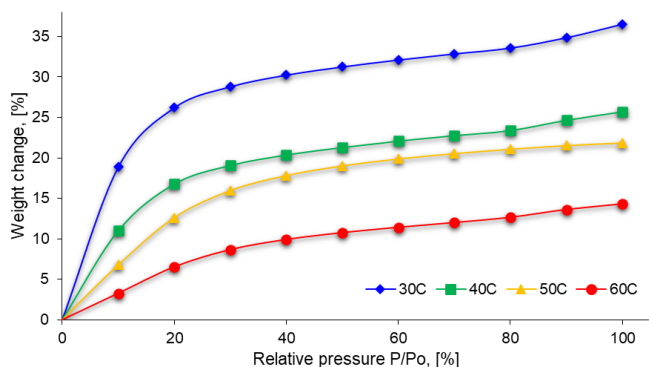


Figure 19 – An illustrative stack depiction of CWZ22 sample adsorption isotherms at different temperatures

Similar to sample WA TONG, both commercial samples CWZ22P and CWZ22 exhibits isotherm of type I. its methanol adsorption uptake decreases with an increase of adsorption temperature. The maximum methanol uptake for both samples was obtained at relative pressure of 1 on the 30 °C isotherm curve with approximate values of 27% and 36% adsorbent weight for CWZ22P and CWZ22, respectively.

### 3.3. Performance takeaways and comparison with in literature activated carbons

Like most activated carbon methanol pairs, all the three studied samples (WA TONG, CWZ22 and CWZ22P) resulted in adsorption isotherms of Type I which is indicative of strong adsorbate adsorbent interactions. The nature of these interactions depends on different parameters such as pore size distribution, surface complexes, the nature of the adsorbate, and isotherm temperature. In a predominantly microporous adsorbent adsorption happens mainly by pore filling mechanism [9]. At four adsorption temperature points of analysis, WA TONG - methanol isotherms exhibit characteristics typical for Type I Langmuir isotherm with subtle differences in the curve gradient rise to the plateau of the isotherm. For activated carbon samples the gradient rise is usually within the relative pressure ( $p/p_0$ ) range of 0.05-0.20 before reaching the inflection point [10]. The plausible explanation for these subtle deviations is the insufficient equilibrium time steps at respective points. Looking at the red adsorption rate curves on respective WA TONG kinetics charts at all three adsorption temperature points, there is inconsistency in peaks pattern relative to the predefined method timestep. The method timestep for all increments

was set at 20 minutes. However, as the adsorption rate is usually higher at the start of the process, the fact that the adsorption curve fails to touch the base in the first three peaks shows the need to increase the step duration for the initial increments to ensure equilibrium is reached. This might have in turn improved the methanol adsorption uptake value at lower relative pressure.

Of the three studied samples, WA TONG exhibits superior sorption performance on all four adsorption temperature points of the analysis. Methanol adsorption uptake for WA TONG sample goes as high as 60 % of adsorbent weight on the 30 °C isotherm at a relative pressure of 1. The characterization analysis of WA TONG sample showed an average porosity and pore volume of about 61% and 0.73  $\text{cm}^3/\text{g}$ , respectively. This falls way below the in literature best performing methanol activated carbon adsorbent – Maxsorb III, whose methanol uptake at 30 °C adsorption temperature can reach up to 1.24  $\text{kg}/\text{kg}$  (124%) at saturation conditions [11]. Maxsorb III is a highly microporous adsorbent with BET surface area of and micropore volume of about 3045  $\text{m}^2/\text{g}$  and 1.7  $\text{cm}^3/\text{g}$ , respectively [12].

However, in literature there are few activated samples with comparable adsorption uptakes to that of WA TONG at the working conditions similar to our experiment. In the study by Henninger et al where the methanol adsorption of six different activated carbons were put to test, sample Carbo Tech A35/1 exhibited an adsorption uptake of 0.58  $\text{kg}/\text{kg}$  (58%) at the saturation conditions and adsorption temperature of 30 °C [13].

## 4. Conclusion

The maximum attained methanol uptake at saturation pressure conditions and adsorption temperature of 30 °C was found to be about 60% for WA TONG, 27% for CWZ22P and 36% for CWZ22. The WA TONG sample performance fell short in comparison to the in literature best performing methanol activated carbon adsorbent – Maxsorb III whose adsorption capacity of 124% at similar working conditions. Subsequently this presents a technical limitation that could be a hindrance for adoption of such samples for commercial use adsorption chiller applications. There might exist a



performance/cost tradeoff for biomass-based activated carbon adsorbents which could lead to wider adoption, hence further investigation study of the WA TONG sample on a pilot scale adsorption chiller could give more clarity on the subject.

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