Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2015.1077348

57 (2016) 16078–16085
July

Taylor & Francis
Taylor & Francis Group

Metal chloride salts in the preparation of activated carbon and their hazardous outlook

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Received 19 January 2015; Accepted 19 July 2015

ABSTRACT

Zinc chloride ($ZnCl_2$) is a well-known activating agent in the preparation of activated carbon, a material that is widely used in the treatment of wastewater. However, the use of $ZnCl_2$ rendering the inevitable aquatic environmental issues due to the toxicity of metal cations. This paper highlights an overview on the latest development of metal chloride-activated carbon with special focus on the characteristics of activated carbon and the hazardous effects of metal chloride salts on human health and environment. The discussion is directed to shed some light on the feasible use of less hazardous metal chloride salts in the preparation of activated carbon.

Keywords: Activated carbon; Activation; Carbonaceous precursor; Hazards rating; Metal chloride salts; Water treatment

1. Introduction

In cellulose pyrolysis, an organic compound with six carbon ring known as levoglucosan is produced resulting in the formation of tar [1]. Some of the pores of resultant carbon are filled or partially blocked with tars rendering its use in adsorption becomes ineffective. In activated carbon preparation, activating agent acts as dehydrating agent to inhibit the formation of tar during pyrolytic decomposition and to increase the carbon yield. Activating agents, such as alkali metals hydroxide, carbonates, metal chloride salts, and some acids can be used in chemical activation of carbon. The frequently used activating agents include KOH, K_2CO_3, Zinc chloride ($ZnCl_2$), and H_2SO_4 [1–3].

The use of $ZnCl_2$ as activating agent has been intensively studied for the preparation of activated carbon from various agricultural wastes—wood apple shell [4], palm shell [5], coffee ground [6], coir pith [7], safflower seed press cake [8], etc. It should be noted, however that zinc cation is a well-known aqueous pollutant. It is toxic to the aquatic organisms and may cause long-term adverse effects to the aquatic environment.

These concerns gradually become a major subject of interest to search for non or less toxic activating agent. It is probable to believe that metal chloride salts having similar characteristics with $ZnCl_2$ such as $FeCl_3$, $CaCl_2$ and $MgCl_2$ are among the potential alternatives to $ZnCl_2$. It is therefore imperative to evaluate their toxic characteristics in the production of activated carbon. The discussion was centred on the

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implications of metal chloride salts to public health and environment without jeopardizing the characteristics of activated carbon produced.

2. Metal chloride salts activation: recent studies

Impregnation ratio (weight ratio of chemical agent to precursor), temperature and period of activation are generally manipulated in the preparation of activated carbon. These parameters affect the physico-chemical characteristics of the resultant activated carbon. Table 1 summarizes the effects of these parameters on the characteristics of metal chloride-activated carbons.

Carbon yield could be regarded as a measure of activated carbon performance. Angin et al. [8] studied the effect of $ZnCl_2$ impregnation ratio and activation temperature on carbon yield. A higher impregnation ratio usually results in a lower carbon yield. Hussaro [5] presented a decrease in the yield of $ZnCl_2$-activated palm shell carbon from 25 to 15% as the impregnation ratio increases from 1 to 3. The activating agent enhances the elimination of light and volatile matter by breaking the aliphatic and aromatic bonds, thus leading to the loss in weight [21]. Angin et al. [8] also reported that activation at 900˚C results in a lower yield of 30% as compared to 75% at 600˚C. At a much higher temperature, the cellulose is converted into graphitic carbon structure due to the decomposition of hemicellulose, splitting off of cellulose and breaking down of lignin structure. Likewise, high molecular volatile matters like tar-containing phenolic compounds are continuously released thus rendering the drop in carbon weight [21–23].

On the other hand, increasing the impregnation ratio and activation temperature usually result in higher specific (BET) surface area and micropore volume of activated carbon [5,8]. This is due to the evolution of volatile matter that enhances the development of pores in char. The creation of new pores increases the BET surface area and the microporosity of activated carbon. In addition, additional pores are created at a higher temperature due to the evolution of high molecular compounds leading to the merging of micropores into mesopores [8,22]. While the excess chemical deposited on the external surface of carbon because of too high impregnation ratio could result in creating wider pores [3].

Boiling point of activating agent is one of the measures to select the activation conditions. At temperature above the melting point, the liquid chemical is mobile and intercalated into the carbon matrix to create pores. Also, the reaction between carbon atoms and vapour chemical species is more significant in widening of the interlayers of carbon [24].

From the previous studies, the use of activating agent generally improves the carbon content of activated carbon. This indicates that the graphitic (aromatic) structure has become dominant [21]. From Table 1, carbon content of safflower seed press cake increased from 49.5 to 76.3% after carbonization and activation with $ZnCl_2$ [8]. Upon activation, rice husk-activated carbon possesses 78% carbon content as compared to 38% in its raw material [14].

From Table 1, $ZnCl_2$ has proven to offer activated carbon with well-developed porosity and high BET surface area for various precursors. Some of the resultant-activated carbons exhibit BET surface area of higher than 1,000 m^2/g [11,12]. $FeCl_3$ and $MgCl_2$ are promising alternatives to $ZnCl_2$ that can generate BET surface area of more than 800 m^2/g [6,16]. Besides, some authors are silence on the measured specific surface area of metal chloride-activated carbon although the adsorption performance was investigated. Sahira et al. [25] reported a maximum methylene blue adsorption of 144 mg/g by $CaCl_2$-activated carbon from Lapsi seed stone. In a different development, KCl was used to activate Jojoba seed residue with iodine adsorption number of 580 mg/g [26]. NaCl is also a potential activating agent. Okeola et al. [17] reported the preparation of NaCl-activated Jatropha fruit pericarp carbon with maximum methylene blue adsorption of 87.7 mg/g.

The size of metal cations of activating agent may be possible to render activated carbon with small and uniform pores. Oliveira et al. [27] reported that due to smaller ionic radius of Fe^{3+} ions (55 pm) compared to that of Zn^{2+} (74 pm), the formation of pores of $FeCl_3$-activated husk centred at approximately 8 Å, while that of $ZnCl_2$-activated husk having part of the pores above 20 Å. Remaining activating agent deposited on the surface of carbon matrix can be removed by washing with HCl solution. The effect of HCl washing also contributes to the evolution of additional pores [3,8]. Thus, smaller size metal ions may act as a template for creating smaller pores [3]. However, this may not happen at high temperatures (600–700˚C) as the gasification of carbon matrix into carbon monoxide and carbon dioxide enlarges the pores. Activating agent also acts as the catalyst to enhance carbon gasification. Hence, the evolution of porosity due to this reaction explains that small ion radius may cause larger pores [6]. For same impregnation ratio of 1, $ZnCl_2$-activated coffee ground displays a greater microporosity of 81%, than $MgCl_2$-(4.76%) and $FeCl_2$-activated coffee ground (32.8%) [6].

Table 2 shows the effect of metal chloride salts on surface functionality of activated carbon.

Table 1
Preparation of metal chloride-activated carbon

					Activation procedure						
Raw material	Carbon content precursor (%)	Carbon content-activated carbon (%)	Agent	Ratio	gas	Temp. (˚C)	time (h)	Yield (%)	Surface area (m^2/g)	Microporosity (%)	Refs.
Wood apple shell[b]	–	–	$ZnCl_2$	10% w/v	–	550	1	42	925	59.2	[4]
					N_2	800	2				
		–	$ZnCl_2$	10% w/v	–	550	1	37	845	71.1	
					–	800	2				
Palm shell[b]	49.9	–	$ZnCl_2$	1	N_2	600	1	25.1	523	53.7	[5]
					N_2	700	2				
		–		3	N_2	700	2	15.2	551	54.9	
					N_2	700	2				
Coffee ground[a]	–	–	$ZnCl_2$	1	N_2	900	1	35.9	977	81.3	[6]
		–	$MgCl_2$	1				25.4	123	4.76	
		–	$FeCl_3$	1				38.6	846	32.8	
Coir pith[a]	–	–	$ZnCl_2$	0.5		700	1	–	910	36.1	[7]
Safflower seed press cake[b]	62.45 (49.50 before pyrolysis)	76.29	$ZnCl_2$	4	N_2	500	–	30	802	72.8	[8]
					N_2	900	1				
		69.87		1	N_2	500	–	42	620	67.6	
					N_2	900	1				
		70.44		4	N_2	500	–	75	249	51	
					N_2	600	1				
Biomass of *Elaeagnus angustifolia* seeds[a]	–	–	$ZnCl_2$	3	–	–	–	–	697	–	[9]
Barley husk[a]	41.16	–	$ZnCl_2$	1.1	N_2	430	0.33	48.5	811	30	[10]
Vetch[a]	–	–	$ZnCl_2$	3	–	240	3	–	1287	–	[11]
Acorn shell[a]		–	$ZnCl_2$	0.5	N_2	600	0.5	13	1289	–	[12]
Maize cob[a]	–	–	$ZnCl_2$	0.15	–	500	1	42.9	11.6	–	[13]
		–		0.05				35.6	702	–	
Rice husk[b]	38.1	78.2	$ZnCl_2$	1.04	N_2	300	0.5	34.2	604	–	[14]
					CO_2	500	1.71				

(*Continued*)

Table 1 (*Continued*)

Raw material	Carbon content precursor (%)	Carbon content-activated carbon (%)	Agent	Ratio	Activation procedure			Yield (%)	Surface area (m^2/g)	Microporosity (%)	Refs.
					gas	Temp. (°C)	time (h)				
Cassave peel[a]	–	–	$ZnCl_2$	0.75	CO_2	700	0.5	–	648	–	[15]
Mango shell[a]	–	–	$MgCl_2$	0.75	–	800	0.5	–	936	–	[16]
Jatropha fruit pericarp[a]	–	–	NaCl	–	–	450	3	39.8	–	–	[17]
Jatropha seed coat[a]	–	–	NaCl	–	–	450	3	35.7	–	–	
Paper sludge[a]	39.52	–	$ZnCl_2$	–	–	600	1	12.4	748	79	[18]
Flame tree[a]	–	–	$ZnCl_2$	0.2–0.4	–	400	1	–	916	–	[19]
Date pit[a]	–	–	$FeCl_3$	1.5	–	700	1	47.1	780	81.7	[20]

[a] One-step activation.

[b] Two-step activation.

According to Yang et al. [28] and Sun et al. [31], the development of surface functional groups is dependent upon type of activating agent, type of precursor and operating conditions. From Table 2, it is observed that the surface oxygen groups of activated carbon are strongly affected by activating agent and type of precursor. However, the concentration of functional groups is not influenced by the increase in impregnation ratio [29]. In other words, the surface functionality of activated carbon is mostly dependent on type of precursor.

Surface oxygen groups are usually formed when the activated carbon is prepared under oxidizing environment. This can be done by exposing the precursor with strong oxidizer such as steam, or the precursor is first treated with reducing agent such as HNO_3 prior to activation. Metal chloride salts, on the other hand, are not reducing agents (or oxidizers). Thus, the evolution pattern of surface oxygen groups could not be clearly observed. In addition, the higher the activation temperature, the higher the propensity of the surface groups to be liberated as they are not stable with temperature.

3. Hazardous properties of metal chloride salts

In general, activated carbon is not toxic and has no significant ecological hazards because of its stable graphitic structure. In addition, the surface functional groups present in activated carbon only contribute towards the removal performance [30]. That is why activated carbon is also used for oral medicinal purposes. Nevertheless, the chemically prepared activated carbon, as opposed to the traditional steam-activated carbon, may prompt toxicities as a result of activators used in its preparation. Inherent drawback of chemical activation is that the residual chemicals remain on activated carbon after washing can leach out during adsorption, thereafter leading to secondary pollution problem [3]. It is therefore imperative to evaluate the hazardous properties and toxicities of activators in the preparation of activated carbon.

Hazard rating (HR) is the score given based on health effect with risk phrases (R-phrases) assigned to each chemical. It is developed by "classification, packaging and labelling" of hazardous chemicals regulation 1997 (CPL regulation 1997) under OSHA; 1 indicates nonhazardous, and 5 indicates most hazardous to health [32].

Hazard material identification system (HMIS) is a numerical HR. It was developed by the American Coatings Association as a compliance aid for the OSHA Hazard Communication Standard. The level of health hazard; 0 indicates no significant risk to health,

Table 2
Effect of metal chloride salts on surface functionality of activated carbon

			Functional groups (mmol/g)			
Precursor	Activating agent	Impregnation ratio	Carboxylic	Lactonic	Phenolic	Refs.
Almond shell	Steam	–	–	–	0.150	[28]
	$ZnCl_2$	3	0.217	0.0481	0.0541	
Grape stalk	$ZnCl_2$	0.5	0.00557	0.0059	0.00615	[29]
		1	0.00537	0.0057	0.00617	
		1.5	0.00547	0.0056	0.00620	
		2	0.00545	0.0057	0.00617	
Arundo donax Linn	$FeCl_3$	1	0.506	0.300	1.45	[30]
	$FeCl_2$	1	0.215	0.413	1.22	
Arundo donax Linn	Steam	–	1.65	1.05	1.65	[31]
	$FeCl_3$	–	1.15	0.78	1.70	

and 4 indicates life-threatening, major or permanent damage from single or repeated overexposure [33]. Both ratings can be used as guide to indicate the effect of chemicals to human health. The R-phrases-based HR are given in Table 3.

Acute effects include acute lethal effects (R20–28), nonlethal irreversible effects after single exposure (R39, R40), corrosive (R34, R35), irritant (R36–38, R41) and sensitizer (R42, R43). While chronic effects include severe effects after repeated or prolonged exposure (R48), carcinogen (R40, R45, R49), mutagen (R46, R40), reproductive hazards (R60–64) and teratogen (R47) [32]. For instance, a chemical labelled with R20/22 and R35 is harmful to inhale and ingest (R20/22), and corrosive to skin (R35). According to CPL regulations, HR for corrosive is higher than that for harmful, so the chemical is classified as corrosive with HR of 4.

Table 4 summarizes the implications of some metal chloride salts to the environment and human health.

Knowing that the boiling point of some activating agents are higher than the activating temperature, residual activating agents that remain on the carbon matrix could prompt inevitable release of possible toxic substances to the environment. Heavy metal such as zinc, lead and copper are known as toxic chemicals. Thus, sources of these heavy metals, namely, $ZnCl_2$, $PbCl_2$ and $CuCl_2$ are very toxic to the aquatic organisms. These chemicals are also hazardous to human health. $FeCl_2$, $FeCl_3$ and $CaCl_2$ are irritant chemicals.

Lethal concentration 50 (LC50) is a standard measure of toxicity of the surrounding medium that could kill half of the test animal population in a specified period. Both $FeCl_2$ and $FeCl_3$ are harmful to some

Table 3
R-phrases-based HR [32]

			Exposure				
			Dermal				
Effect		Inhale	Skin	Eye	Ingest	Not specified	Hazard rating (HR)
Very toxic	Acute	R26	R27	–	R28	R39	5
	Chronic	–	–	–	–	–	–
Toxic	Acute	R23	R24	–	R25	R39	4
	Chronic	–	–	–	–	R48, R39	4
Harmful	Acute	R20	R21	–	R22	R40	3
	Chronic	–	–	–	–	R48, R40	3
Corrosive	Acute	–	R35	–	–	–	4
		–	R34	–	–	–	3
Irritant	Acute	R37	–	R41	–	–	3
		–	R38	R36	–	–	2

Table 4
Effects of metal chloride salts to human and the environment [34–53]

Salt	r_{ion}[a] (pm)	Boiling point (°C)	R-phrase	HR	HMIS	Effect to human	Effect to environment
$CaCl_2$	100	1935	R36	2	2	Irritating to eyes; damage to heart and cardiovascular system	The products of degradation are less toxic than the product itself
$PbCl_2$	119	950	R20/22 R33 R61 R62 R50/53	3	3	Harmful by inhalation and ingestion; damage to kidney, central nervous system, blood forming organs; harm to the unborn, impaired fertility	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment; the products of degradation are more toxic
KCl	138	1420	R36	2	1	Irritating to eyes; damage to eyes, blood, cardiovascular system	The product itself and its products of degradation are not toxic
LiCl	76	1382	R22R36/38 R61 R62	2	2	Harmful if swallowed; irritating to skin and eyes; harm to the unborn, impaired fertility; damage to central nervous system, cardiovascular system; teratogenic	The products of degradation are less toxic than the product itself
$CuCl_2$	73	993	R25 R36/37/38 R50/53	4	3	Toxic if swallowed; irritating to eyes and skin; damage to kidneys, lungs, liver, mucous membranes	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment; the products of degradation are more toxic
$ZnCl_2$	74	730	R34 R50/53	3	3	Corrosive to skin and eyes; damage to kidneys, pancreas; possible mutagenic effects	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment; the products of degradation are less toxic than the product itself
$FeCl_2$	61	1023	R22 R34 R36/38	3	3	Harmful if swallowed; irritating to skin and eyes; damage to lungs, mucous membranes	Harm to fish, the products of degradation are more toxic
$FeCl_3$	55	315	R36/38	2	3	Irritating to skin and eyes; damage to lungs, mucous membranes	Harm to fish, the products of degradation are more toxic
$MgCl_2$	72	1412	–		2	Slightly hazardous in case of skin and eye contact, ingestion and inhalation	The products of degradation are more toxic
NaCl	102	1413	–		1	Slightly hazardous in case of skin contact, ingestion and inhalation	This product has no known eco-toxicological effects under normal use; it is readily biodegraded, the product itself and its products of degradation are not toxic

[a] Cationic radius according to effective ionic radius [54].

aquatic organism due to the iron properties with LC50 of 4–6 mg/L. The degradation product of $CaCl_2$ is less toxic to the environment. $MgCl_2$ is slightly hazardous to human health but its degradation product is more toxic to the environment. KCl and NaCl are slightly irritant chemicals in case of contact, but both of them are not toxic to the environment under normal use. In short, KCl and NaCl are the least toxic activating agents among the metal chloride salts, and could be of interest as the potential substitutes to the commonly used, but toxic $ZnCl_2$.

4. Concluding remark

Generally, higher impregnation ratio and higher activation temperature facilitate the improvement of specific surface area and micropore volume, but result in lower activated carbon yield. However, further increase in impregnation ratio as well as temperature lead to a sharp decline in specific surface area and micropore volume due to pores enlargement. Apart from the process conditions, the performance of activated carbon also depends on the types of activating agent. It is believed that smaller size of cationic radius renders smaller pores at temperature below 600˚C. $ZnCl_2$ is a widely used activating agent in the preparation of activated carbon with high specific surface area, but its aqueous toxicity is of considerable concern in the present studies. Less hazardous metal chloride salts with similar attributes as that of $ZnCl_2$ would be promising alternatives to $ZnCl_2$. $FeCl_3$, $MgCl_2$, $CaCl_2$, KCl and NaCl are among the potential activating agents that have been reported in the literature. Among others, KCl and NaCl are the least hazardous to public health and so to the environment. Yet, further studies would be needed to ascertain the suitability of these activating agents in the preparation of activated carbon.

Acknowledgement

Authors are thankful to the Ministry of Education Malaysia and Universiti Teknologi Malaysia for the awards of Fundamental Research Grant Scheme No. 4F305 and Flagship #03G07. L. Lin-Zhi acknowledges the support of MyBrain15 by the Ministry of Education Malaysia.

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