



# Studies On Cationic Matrices Blended With Sulphonated Morinda CitrifoliaL., Carbon

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## ARTICLE INFO

## ABSTRACT

Resorcinol – formaldehyde resin (RFR) was prepared and blended with sulphonated charcoals (SCs) prepared from a plant material. Composite ion exchange resins (IERS) were prepared by varying the amount of SCs (10-50%w/w) in the blends. All the important physico - chemical properties and Instrumentation techniques such as FT-IR, TGA-DTA and SEM have been analysed. Composites up to 30% (w/w) blending retain almost all the essential characteristics and Cation Exchange Capacity (CEC) of the original PFR. It is concluded that blending of PFR by SCs will reduce the cost of IERS.

**Keywords:** Resorcinol – formaldehyde Resin – Sulphonated Morinda CitrifoliaL. Charcoal – Cation Exchange Capacity – Composite resins – Ion Exchangers

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## 1.0 INTRODUCTION

Metal toxicity or metal poisoning is the toxic effect of certain metals in certain forms and doses on life. The non-biodegradable / toxic heavy metal ions will penetrate into the environment and may eventually affect all the forms of life including human beings. Many heavy metal ions will enter into water sources, because of the prevailing unproductive treatment and disposal methods. Considering the industrial wastewater treatments, **ion exchange** is an appropriate technique for the removal and recovery of heavy metal ions, as it is employed in the separation and concentration of ionic materials from liquids [1]. Many commercial resins (CRs) owe their origin to petroleum products and their cost is increasing continuously due to the scarcity of petroleum reserves. Difficulty also exists in its procurement. Therefore, there is an urgent need to find out the new low-cost ion exchange resins (IERS) and reduce the cost of IERS. Such types of low-cost ion exchangers could be obtained by blending the IER with SC obtained from cheaper and freely available plant materials containing phenolic groups in it. Attempts have been made in earlier studies to prepare cheaper condensate cationic resins (CRs) by partially blending the macroreticular phenol-formaldehyde sulphonic acid resin viz., the cationic matrices (CMs) with SCs prepared from natural products like, coal [2], saw dust [3], spent coffee [4], cashew nut husk [5], wheat husk [6], turmeric plant [7], spent tea, gum tree bark [8], Accacianilolica [9], Egyptian bagasse pith [10], creativanurvalabuch. Ham [11], Terminalia bellerica Roxb [12] and Solanum xanthocarpum [13]

Similarly, the research studies have also been reported in which substituted phenol-formaldehyde resin matrix was blended with sulphonated charcoal obtained from coconut fibre (resorcinol-formaldehyde) [14] coconut shell (p-cresol-formaldehyde) [15] and curcuma longa (resorcinol-formaldehyde) [16]

Ion exchange process finds a valuable place in the treatment of wastewater containing heavy metal ions discharged from electro-plating and other industries. In addition, ion exchange is a convenient way to concentrate, remove and recover the ions of valuable metals. Special processes using selective IERS are also available in literature for the removal and recovery of precious metals [17].

The aims and objectives of the present work are to determine the optimum conditions for the preparation of condensates obtained by blending CMs of RFSAR with various % (w/w) of sulphonated Morinda citrifolia L. carbon (SMCC), to characterise the new condensate of sulphonated cationic

exchangers (CEs) and to estimate the column / cation exchange capacity (CEC) or ion exchange capacity (IEC) for some selective metal ions. The effect of particle size, chemical and thermal treatment of IERs on CEC has been studied. The regeneration level of  $Pb^{2+}$  loaded resin is studied with NaCl (brine) solution.

## 2.0. EXPERIMENTAL

### 2.1. Materials

The plant material used in the present study was *Morindacitrifolia* L., (Family- Rubiaceae in English- Indian Mulberry and in Tamil-Manjanathi and in Hindi: Bartundi). This is a plant material freely available in Tamil Nadu, India. Resorcinol and formaldehyde used were of Fischer reagents (India). AR grade of con. sulphuric acid (Sp.gr. = 1.82) was used. The plant material was locally collected, cleaned, dried and cut into small pieces of about 0.5 cm length. The other chemicals and reagents were of chemically pure grade (AnalaR) procured from SD fine chemicals, India.

### 2.2. Methods

*Morindacitrifolia* L. barks (500g) have been carbonised and sulphonated by con. sulphuric acid, washed to remove excess free (tested with  $BaCl_2$  solution) acid and dried at 70°C for 12 h. It was labeled as SMCC. The method followed was similar to the reported one for preparing SCs from plant materials [6-8].

Resorcinol - Formaldehyde resin (RFR) was prepared according to the literature method [3, 6 - 8]. It was then ground, washed with distilled water and finally rinsed with double distilled (DD) water to remove excess free acid, dried, sieved (210 - 300  $\mu$ ) using Jayant sieves (India) and preserved for characterisation [3,6-8,17]. The resorcinol-formaldehyde resin (100 % pure resin) was labeled as RFR.

The condensates of RFR with various % (w/w) of SMCC were obtained as per the method reported in literature [3,6 - 8]. The products with 10, 20, 30, 40 and 50% (w/w) of SMCC in the condensates, respectively are labeled as MC1, MC2, MC3, MC4 and MC5. A separate sample of SMCC (100 % pure) was also subjected to the characterisation studies.

### 2.3. Characterisation of Samples

FT-IR spectral data of pure resin (RFR), condensate obtained by blending of RFR with 30% (w/w) of SMCC and SMCC (100 % pure) were recorded with a PerkinElmer, Make: Spectrum RX I with KBr pellets. To establish the thermal degradation of the samples, TGA and DTA traces were obtained for RFR condensate and pure SMCC were obtained by using Perkin Elmer Pyris 1 TGA. The surface morphologies of synthesized IERs were studied using a scanning electron microscope (SEM, JEOLJSM 6390).

Samples were ground and sieved into a size of 210 - 300  $\mu$  using Jayant sieves (India). This was used for further characterisation by using standard procedures [3, 7, and 8,] to find out the values of absolute density (wet and dry density in water and toluene, respectively), percentage of gravimetric swelling and percentage of attritional breaking. The solubility of these samples was also tested in various organic solvents and inorganic reagents. Characterisation of samples was carried out, following the methods reported in literature [3, 6-10]. The values of cation exchange capacity (CEC) were determined by using standard titration techniques as per the literature method [18-21]. The effect of particle size, chemical and thermal stability of the RFR and condensates on CEC was also determined [22].

After the exchange of  $H^+$  ion by the metal ions, the regeneration level of the condensates loaded with a metal ion (especially  $Mg^{2+}$ ) were determined by using NaCl (brine) solution.

## 3.0. RESULTS AND DISCUSSION

### 3.1. SYNTHESIS OF CONDENSATES

The experimental (obs) and theoretical (cal) composition of SMCC in the condensates (MC1 – MC5) are in good agreement with each other (Table 1). The results are similar to those obtained by Sharma et al. [2]. This indicates that the preparative methods adopted for the synthesis of RFR and its condensates (MC1 – MC5) are more reliable and reproducible.

### 3.2. CHARACTERISATION BY INSTRUMENTAL STUDIES

FT-IR spectral studies are used to confirm the stretching frequencies of various functional groups and to identify the ion exchangeable groups [23] present in IER. The appearance of absorption bands at 1022-1036  $cm^{-1}$  (S = O str.) 1158-1161  $cm^{-1}$  ( $SO_2$  sym. str.) and 576-599  $cm^{-1}$  (C – S str.) in RFR (pure resin), condensate MC3 and pure SMCC (100 %) confirm the presence of sulphonic acid group (Fig.1). The relevant data are given in Table 2. The data also indicate the presence of phenolic groups.

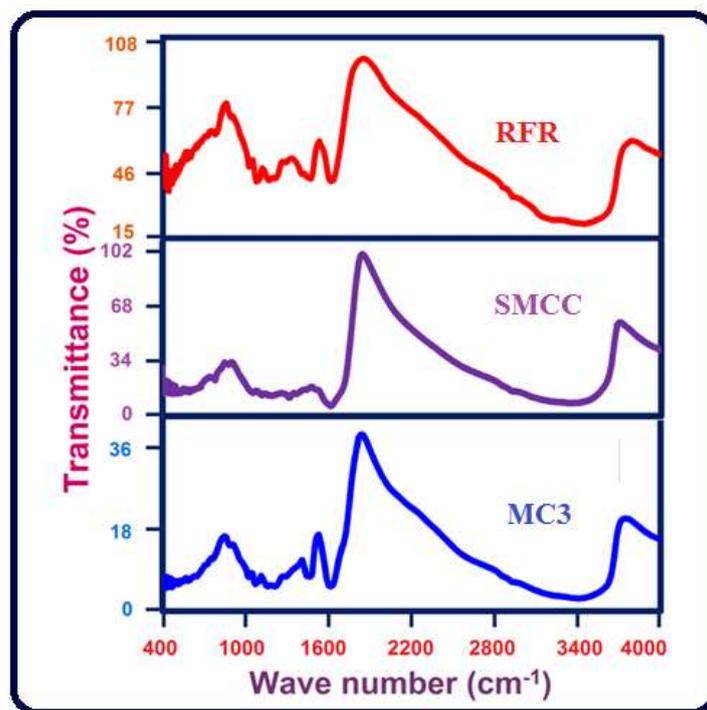
**Table 1 Amount of reagent used and yield of RFR and condensates (MC1–MC5)**

Sample	% of SMCC in IER	Amount of reagents used			SMCC (g)	Yield (g)	% of SMCC in IER(Obs)
		Resorcinol (g)	HCHO (mL)	Con. H <sub>2</sub> SO <sub>4</sub> (mL)			
RFR	0	10.0	11.5	12.5	0	16.00	0.00
MC1	10	10.0	11.5	12.5	1.77	17.24	10.03
MC2	20	10.0	11.5	12.5	4.00	18.18	22.00
MC3	30	10.0	11.5	12.5	6.86	21.40	32.05
MC4	40	10.0	11.5	12.5	10.67	25.84	41.29
MC5	50	10.0	11.5	12.5	16.00	31.06	51.51
SMCC	100	--	--	--	--	--	100

The appearance of a broad absorption band at 3273-3345cm<sup>-1</sup> (bonded -OH str.) indicates the presence of phenolic and sulphonic -OH (due to -SO<sub>3</sub>H) groups in the samples. The appearance of an absorption band at 1601-1621 cm<sup>-1</sup> (C-C str.) confirms the presence of aromatic ring in RFR, condensate MC3 and pure SMCC. The absorption band at 1460 – 14870 cm<sup>-1</sup> (-CH<sub>2</sub>- def.) confirms the presence of -CH<sub>2</sub> group in the samples. The weak absorption band at 868 –890cm<sup>-1</sup>(-CH def.) noted in samples indicate that the phenols are tetra substituted.

**Table 2 FT-IR spectral data of RFR, condensate MC3( 30% (w/w) of SMCC) and SMCC (in cm<sup>-1</sup>)**

Group	PFR	Composite MC3	Pure SMCC
S = O str.	1022	1022	1036
SO <sub>2</sub> sym. str.	1161	1160	1158
C – S str.	599	590	576
Bonded OH str.	3273	3345	3312
CH <sub>2</sub> – def.	1465	1460	1487
C – C str.	1612	1601	1621
C - H def.	868	890	868



**Fig. 1 FT-IR Spectra of RFR, SMCC and MC3**

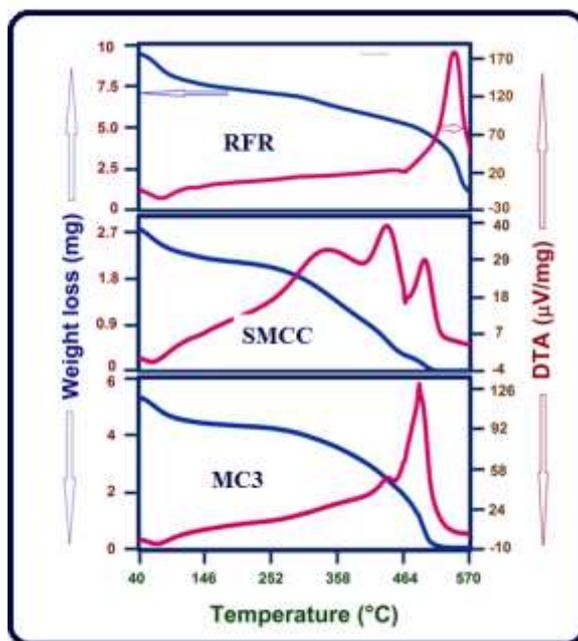
Thermo-gravimetric analysis (TGA) has been widely used for rapid assessment of the thermal stability of various substances [20]. TGA curves shown in Fig.2 reveal that upto 70°C there is 7% loss in weight for RFR, and 8.05% for MC3 and 9.5 % for SMCC respectively. This is due to the loss of moisture absorbed by all IERs. At 450°C there is 42% weight loss in RFR, 48% loss in weight in MC3 and 62% loss in weight in SMCC at 400°C were obtained. This may due to the thermal degradation of IERs. Thermal studies indicate that the IERs are thermally stable up to 100 °C.

Two endothermic peaks are obtained in RFR, approximately at 70°C and at 450°C, respectively (Fig.2). At 70°C, the presence of broad peak indicates the dehydration process of RFR. A peak at 450°C indicates the

chemical changes, which occur due to thermal degradation of RFR, and reflects approximately 42% weight loss in RFR.

DTA curve of MC3 (Fig.2) shows that, the same two types of endothermic peaks are also obtained at 70°C and at 450°C, respectively similar to RFR. Again, the first broad peak indicates the dehydration of MC3 and second moderate sharp peak indicates the chemical changes, which occur due to thermal degradation of MC3 and reflects approximately 48% weight loss in MC3

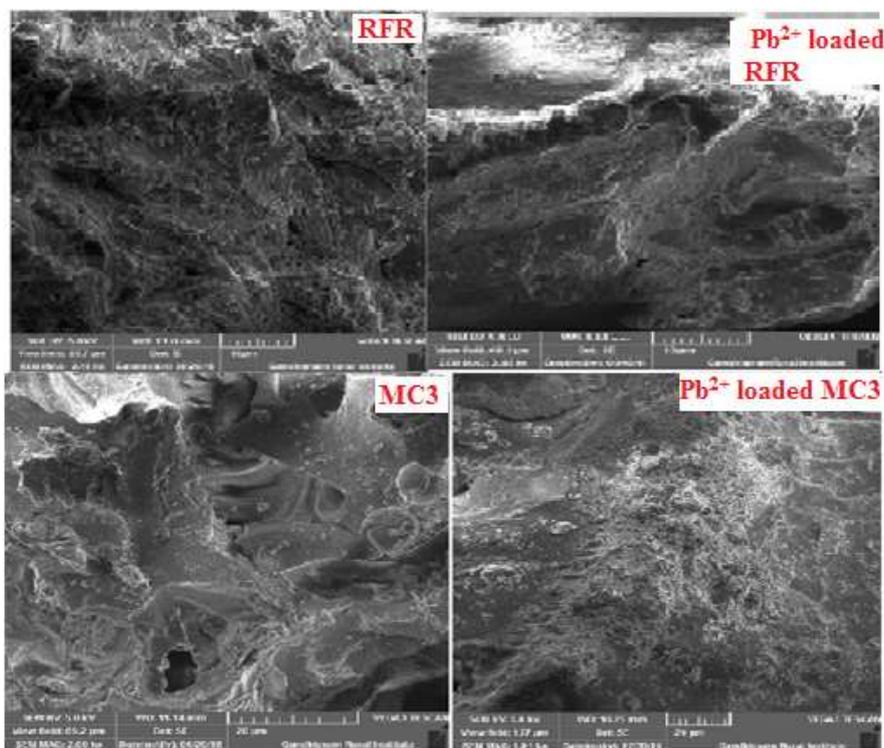
DTA curve of SMCC (Fig.2) shows that, the same two types of endothermic peaks are also obtained at 70°C and at 400°C, respectively similar to RFR. Again, the first broad peak indicates the dehydration of SMCC and second moderate sharp peak indicates the chemical changes, which occur due to thermal degradation of SMCC and reflects approximately 62% weight loss in SMCC.



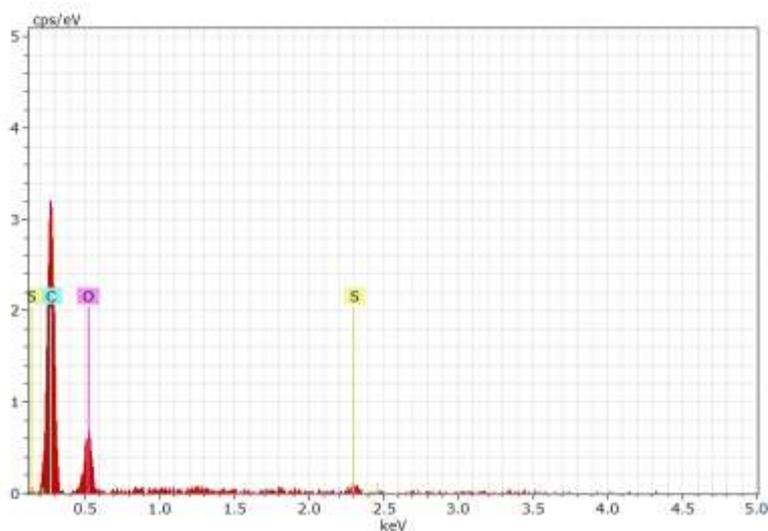
**Fig.2 TGA and DTA traces of RFR, SMCC and MC3**

From the TGA-DTA data conclude that the thermal degradation temperature increases from RFR to MC3 and then to SMCC. Hence the condensate resin MC3 is more thermally stable compare with parent resin RFR. This may due to the filling of SMCC particles into the voids of the condensate resin MC3. It is also concluded that the limiting temperature for the safer use of all IERs was 100°C, since the IERs degrade thermally after 100°C.

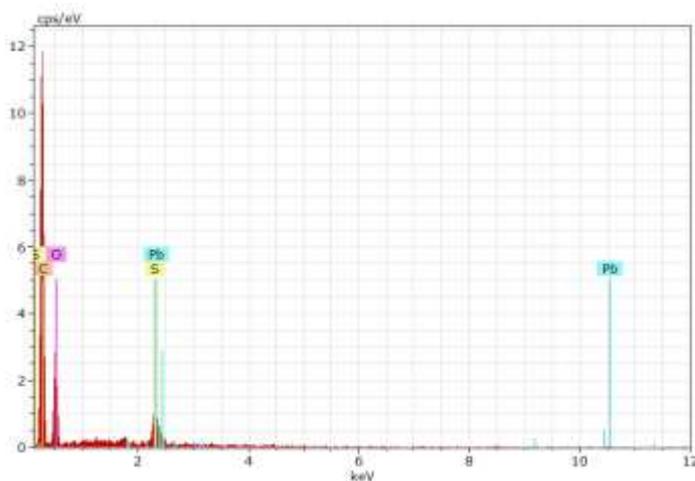
SEM photos of RFR and condensate of MC3 with different magnification are given Fig.3. SEM photos reveal that all these samples are macroporous in nature. The high macroporous carbon obtained from *Morindacitrifolia* L form the reservoir in which the phenol-formaldehyde sulphonic acid particles are deposited. Hence, the pore diameter decreases in MC3.



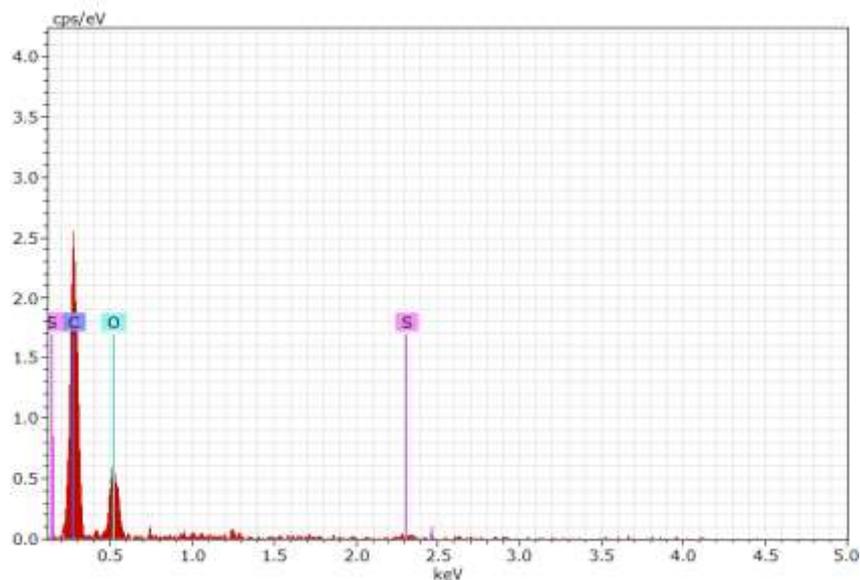
**Fig.3 SEM images of RFR and MC3 with different magnification**



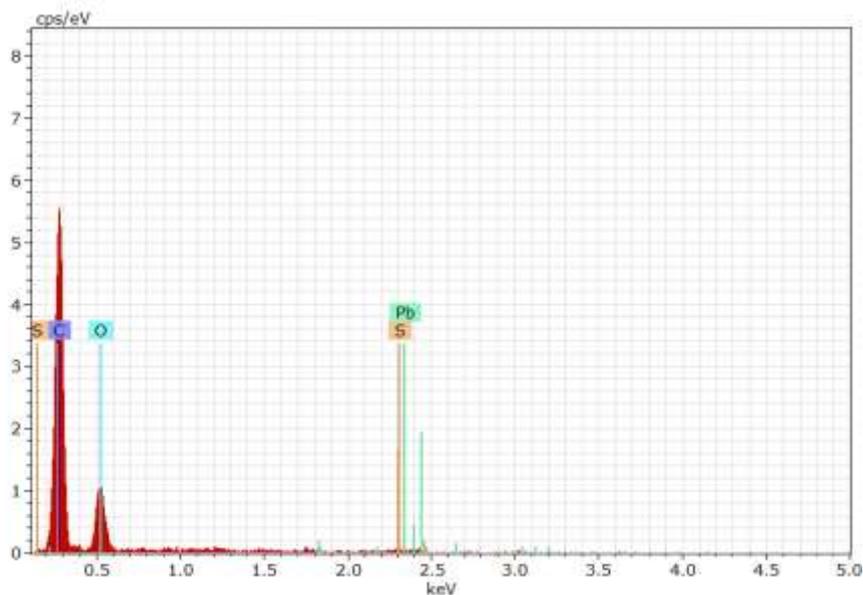
**Fig.4 a EDAX diagram of RFR,**



**Fig.4 b EDAX diagram of lead loaded RFR,**



**Fig.4 c EDAX diagram of MC3,**



**Fig.4 d EDAX diagram of lead loaded MC3,**

Purity of the prepared samples was studied by EDX technique and the results for the IERs are shown in Fig 4a, 4b, 4c and 4d respectively. The peaks are clearly related to C, O, S and Pb elements. Moreover, the EDX spectra of lead loaded RFR and MC3 demonstrate that the peaks corresponding to S along Pb elements which suggest that the  $Pb^{2+}$  ion binding with sulphonic acid group in the ion exchange resin and also noted that no foreign elements present in the compound.

### 3.3. PHYSICO – CHEMICAL CHARACTERISTICS

The absolute density (Table 3) values in both hydrated (in water) and dehydrated (in toluene) states decrease steadily from RFR (100 % pure resin) to 50 % (w/w) SMCC in condensate (MC5) and then finally to SMCC (100 % pure SC). This indicates that RFR and the condensates (MC1 – MC5) are more closely packed [3-10]. It is found that the absolute density of 100% SMCC possess only, 51.51 % and 49.60% of density of RFR in hydrated (wet) and dehydrated (dry) states, respectively. This indicates that SMCC also has closely packed structure [17] similar to condensate.

The values of density of condensates in dry and wet states depend upon the structure of the basic resin unit, its degree of cross-linking and ionic form [23]. Hence, high-density values are obtained for these IERs. The values of density (both wet and dry forms) presented in Table 3, indicate the high degree of cross-linking, and hence the condensate resins are suitable for making columns for treating polar and non-polar effluents. Moreover, the wet and dry density values are close to each other, which indicate that the IER samples may be macro porous in nature.

**Table 3 Physico-chemical properties of RFR, SMCC and condensates (MC1- MC5)**

IERs	% of SMCC in IER	Density (g/mL)		Percentage	
		Wet	Dry	Gravimetric swelling	Attritional breaking
RFR	0	2.52	2.64	88.08	9.45
MC1	10	1.92	1.98	77.36	12.65
MC2	20	1.84	1.88	71.25	17.54
MC3	30	1.72	1.74	67.54	21.26
MC4	40	1.41	1.52	59.23	28.78
MC5	50	1.34	1.41	51.25	31.25
SMCC	100	1.25	1.36	42.88	48.84

From the data given in Table 3, it is clear that there is no considerable decrease in absolute density in both hydrated (wet) and dehydrated (dry) states upto 30% (w/w) blending of SMCC with RFR. In the condensates, indicating that they also have similar closely packed structures with high degree of cross-linking and hence they are suitable for making ion exchange columns for polar and non-polar liquids of high density [22].

The gravimetric swelling percentage (Table.3) decreases from RFR (88.08%) to SMCC (42.88%). Condensates have intermediate values of gravimetric swelling (%). It indicates that the values of gravimetric swelling (%) for RFR, and condensates are not as high as compared to conventional gel type IERs, indicating rigidity in the matrix and therefore the pores of condensates are of non-gel type and macro reticular [17]. SMCC has a swelling capacity of only 48.68 %, as compared to that of RFR. This extremely low value of percentage gravimetric swelling may be due to certain rigidity in the matrix of the resin. The blending of RFR with 30% (w/w) of SMCC has the gravimetric swelling value as 76.68 % of that of RFR, thus decreasing 21.32% of swelling compared to that of RFR ( 100 % for pure resin).

The decrease in % gravimetric swelling is attributed due to the loss of polarity and porosity in condensates. Thus, the condensates may prove to be highly suitable where they are required to withstand a large osmotic shock during its application for the removal of metal ions.

The values of percentage of attritional breaking (Table.3) represent the stability of the IER, which increase from RFR to SMCC. The data reveal that the mechanical stability is good upto 30% (w/w) blending of SMCC (21.26%) with RFR. This observation also shows the possibility of formation of resin in the capillaries of the sulphonated carbon (SMCC) particles [6-8].

The solubility of the IER samples in various organic solvents and inorganic reagents reveals that RFR, condensates and STMC are practically insoluble in almost all the solvents and reagents, except in 20 % (w/w) NaOH. The insolubility of the resin samples even in the highly polar trichloroacetic acid express its rigidity i.e., high degree of cross-linking within the molecules. So it may be concluded that RFR, and the condensates are having high degree of cross-linking in the sample structure i.e., the basic polymer unit has mostly high molecular weight fractions or at least the absence of very low molecular weight fractions in them [17]. Hence, IERs can be used as ion exchangers for treating non-aqueous effluents also [9]. At the same time, the samples are partially soluble in 20% (w/w) NaOH solution, which indicates the presence of phenolic groups in them. Hence, these ion exchange materials cannot treat the industrial effluent having high alkalinity (above pH 7).

### 3.4.CATION EXCHANGE CAPACITY

**Table 4 Cation exchange capacities of RFR, condensates (MC1-MC5) and SMCC for selective metal ions (0.1 M ) at 303 K**

IERs	% of SMCC	Cation Exchange Capacity m mol/g						
		Na <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Zn <sup>2+</sup>	Cu <sup>2+</sup>	Cd <sup>2+</sup>	Pb <sup>2+</sup>
RFR	0	0.799	1.701	1.545	1.590	1.682	0.845	1.832
MC1	10	0.778	1.684	1.212	1.456	1.584	0.745	1.751
MC2	20	0.645	1.521	1.102	1.368	1.512	0.664	1.612
MC3	30	0.636	1.441	1.078	1.288	1.474	0.532	1.563
MC4	40	0.624	1.335	0.845	1.141	1.265	0.454	1.456
MC5	50	0.614	1.212	0.726	1.112	1.158	0.351	1.324
SMCC	100	0.032	0.781	0.612	0.714	0.812	0.021	0.723

Cation/ Column exchange capacity (CEC) or Ion exchange capacity (IEC) values given in Table 4 indicate that, the CEC value decreases when the content of SMCC ( %w/w) in RFR (Fig.5). The relative ion exchange capacity (IEC) of individual metal ions depends upon the atomic radius or atomic number [24]. At the same time, the CEC value also depends upon the valence of the ions, the concentration of metal ion in the influent solution and the anionic part of the metal salt. Inter ionic forces of attraction between anions and cations, which also plays a vital role in deciding the CEC values of metal ions for a given metal salt solution [25,26]. It is found that CEC values of IERs obtained by blending RFR with 30 % (w/w) of SMCC in the range of 79.55% to 85.31% for various metal ions viz., Cu<sup>2+</sup>, Zn<sup>2+</sup>, Pb<sup>2+</sup>, Cd<sup>2+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, and Na<sup>+</sup> ions.

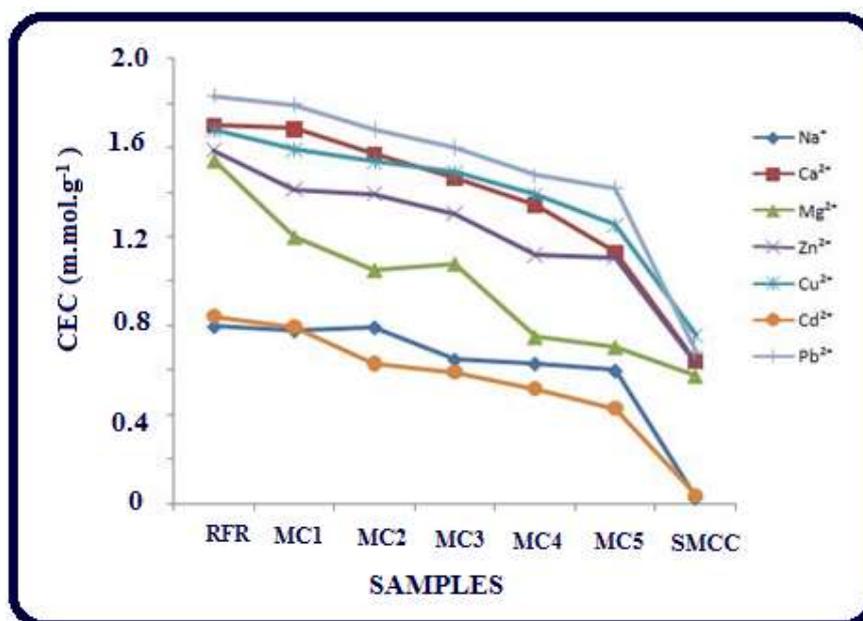


Fig. 5 Cation Exchange Capacities of H<sup>+</sup> form of RFR, condensates and SMCC for various metal ions at 30°C

Hence, it may be concluded that RFR could be blended upto 30% (w/w) of SMCC and the condensates thus obtained could be new / novel and cheap IERs, which could be used for water and wastewater treatment especially for the removal of metal ions. It is also useful in industrial applications, especially for the treatment of industrial effluent containing metal ions or desalination purpose.

From the CEC data given in Table 4.4, the CEC values of IERs are found to decrease in the following order:



The selectivity order of metal ions i.e., orders of CEC value also depends upon the ionic potential and the hydrated atomic radius of the metal ions in solution [26]. The order of exchange affinities of various metal ions is not unique to ion exchange system. Only under dilute conditions Hofmeister or lyotropic series [23] is obeyed. However, under high concentration it is different [23]. It is equally important to note that the relative behaviour of these ions for other ionic phenomena deviate the affinity order under the same conditions [27]. The observed order in the present study is different from that of the Hofmeister or lyotropic series [23]. This may be due to the concentration of the influent metal ion solutions, which is relatively high and also due to the selectivity of the metal ions.

The percentage value of CEC of condensates MC1 - MC5 for exchange of H<sup>+</sup> ions with Cu<sup>2+</sup>, Zn<sup>2+</sup>, Pb<sup>2+</sup>, Cd<sup>2+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, and Na<sup>+</sup> ions in 0.1M solutions is about 58.-64% , for pure commercial (SD's) resin (100%). Nearly 60 % of CEC was retained by these condensates as compared to that of the pure commercial resin. This indicates that the condensates can partially replace commercial IERs in making the ion exchangers for industrial applications.

In order to test the chemical stability of RFR and condensates, the samples were boiled with 20% (w/v) NaOH, benzene and 1M HCl for an hour. The CEC of the resulting treated samples was determined. The treated samples possess CEC almost similar to that of the untreated resins for all the metal ions. This indicates that the resin samples are chemically stable, (Table 5). In order to test the thermal stability of RFR and condensates, the samples were heated to 100 °C in an air-oven for an hour.

Table 5 Chemical and thermal effects on CEC of RFR and its condensates for exchange with 0.1M Pb<sup>2+</sup> ions at 303K

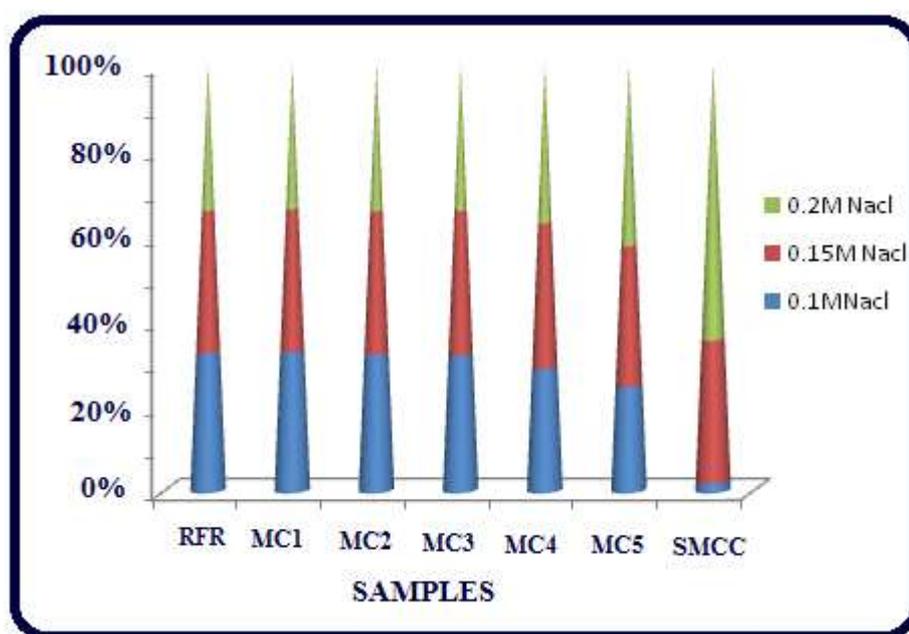
Reagents	Cation Exchange Capacity (m. mol. g <sup>-1</sup> )					
	RFR	MC1	MC2	MC3	MC4	MC5
CEC (of untreated)	1.545	1.189	1.071	1.088	0.814	0.726
20%(w/v) NaOH	1.395	1.092	0.931	0.984	0.752	0.664
Benzene	1.483	1.126	0.984	1.084	0.845	0.741
1M HCl	1.512	1.135	1.074	1.014	0.812	0.722

Although the samples lose their weight initially on heating upto 100°C, the weight lost is regained after cooling for 24 h and exposing it to air. Beyond that temperature (100°C), the thermal degradation is noted to be nearly 6-9% at 120°C. This indicates that the IERs are thermally highly stable upto 100°C. Their CEC values have been determined and also found to be close to that of the untreated IERs.

**Table 6 Effect of particle size of RFR and condensate with 30%(w/w) of SMCC (MC<sub>3</sub>) on CEC at 303K**

Sample	Particle size	Cation exchange capacity mmol/gm				
		Na <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Cu <sup>2+</sup>	Pb <sup>2+</sup>
RFR	<210μ	0.723	1.624	1.486	1.613	1.781
	210-300 μ	0.799	1.701	1.545	1.682	1.832
	300-500 μ	0.654	1.582	1.423	1.547	1.721
	>500 μ	0.628	1.401	1.390	1.393	1.698
MC <sub>3</sub>	<210μ	0.584	1.412	0.874	1.426	1.348
	210-300 μ	0.674	1.523	1.023	1.521	1.466
	300-500 μ	0.521	1.348	0.775	1.394	1.298
	>500 μ	0.412	1.098	0.654	0.961	1.154

CEC values given in Table 6 indicate that the IERs with particle size of <210 micron are fine, 300 - 500 micron and > 500 micron are coarse, as to cause very low ion exchange capacity compared to that of IERs of 210 - 300 micron particle size. Hence, for the effective IEC, the particle size of the IER should be maintained and recommended particle size is 210 - 300 micron.

**Fig.6 Regeneration level for RFR, condensates (MC<sub>1</sub> – MC<sub>5</sub>) and SMCC by using NaCl solution of various concentration after exchange with Mg<sup>2+</sup> ions at 303K**

Forty mL of 0.20M NaCl (brine) solution effectively regenerates all the condensates, RFR and SMCC, after the exchange with the Mg<sup>2+</sup> ions. Most of the commercial IERs are in Na<sup>+</sup> form and hence 40 mL of 0.20M NaCl could be used as a regenerating agent for every 2g of the resin (Fig.6).

### 3.5. CONCLUSION

It is concluded from the results of the present study that RFR could be blended upto 30% (w/w) of SMCC, without affecting its spectral, thermal and physico-chemical properties. CEC values of these CERs are estimated. The effect of particle size on CEC, its regeneration level by using NaCl solution has been studied. CEC values of various metal ions of condensates upto 30% (w/w) SMCC are found to be very close to the RFR. Hence, blending of RFR with STAC to get condensates will definitely lower the cost of IER.

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