



# Characterization of new and fouled SWRO membranes by ATR/FTIR spectroscopy

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## Abstract

Attenuated total reflection (ATR)–Fourier transform infrared (FTIR) spectroscopy was used to characterize the chemical nature of fouling species on different spiral wound membranes operated at Al-Jubail SWRO plant. Three membranes from different companies operated for a period of 1 year were opened for autopsy, and difference spectra were obtained for the fouled membrane from virgin membranes. The spectra of the foulants were easy to distinguish from the spectra of membrane material. The results showed that the membrane surface of the feed end element was more heavily fouled than the brine end of the element. ATR/FTIR indicated a distinct enhancement to the amide I ( $1650\text{ cm}^{-1}$ ) and amide II ( $1550\text{ cm}^{-1}$ ) bands indicative of protein, and the C–O stretching band associated with polysaccharides is visible near  $1040\text{ cm}^{-1}$ . The deposits removed from the surface of membranes showed distinctive bands at  $3270$  and  $1080\text{ cm}^{-1}$  associated with amino group/polysaccharide.

**Keywords** ATR/FTIR spectrometry · Fouling · SWRO membrane · Membrane characterization

## Introduction

Membrane technology is the most widely used and accepted around the world for the treatment and improvement of various water resources. Advancements in membrane materials and other aspects of membrane desalination technology have lowered overall operating costs, which have ultimately reduced the cost of implementing public water quality improvement programmes. Membrane processes are very effective in removing many harmful constituents in water and are deemed the most appropriate water treatment technology for many years. There are different types of membrane desalination processes used in the potable water treatment field. A particular process should be selected based on the most cost-effective way to remove constituents in the feed water to meet the water quality goals. Reverse osmosis

(RO) membranes are found to be more effective in removing majority of constituents. The pore size of a RO membrane is so small that it functions primarily by diffusion instead of filtration or adsorption (Jim 1995). Most of the membranes applicable to water treatment are prepared from synthetic organic polymers and have been classified into cellulosic or non-cellulosic membrane types. The cellulosic types include cellulose diacetate, cellulose triacetate and blends of these. The non-cellulosic types include polyamides (both aliphatic and aromatic), polyurea, polysulfone, etc. (Lozier and Bergman 1990). Manufacturers produce different types of membranes designed for various types of feed water. RO membranes can be classified by cross-sectional form into (1) asymmetric (2) composite. Asymmetric membranes are composed of a single polymer type and contain a thin dense skin that serves as the active desalination barrier layer. Thin-film composite membranes are formed by depositing a very thin polymer film which acts as the active barrier layer onto the surface of a different polymer that serves as the porous support layer (Lozier and Bergman 1990). In spite of several advantages of recently manufactured membranes, composite membranes experienced substantial membrane fouling and/or membrane degradation, due to improperly designed or poorly operated RO system, resulting in the decrease in productivity and also rejection. Membrane fouling could be

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either chemical or biological. Chemical fouling of membrane is due to particulate, colloidal matter, silica, oxides of metals and mineral scales, viz.  $\text{CaCO}_3$ ,  $\text{CaSO}_4$ ,  $\text{Mg}(\text{OH})_2$ ,  $\text{Fe}(\text{OH})_3$ ,  $\text{FeCl}_3$  and organic matter (David and Rehman 1990; Lepore and Ahlert 1988; Vaugham 1989; Farooque et al. 1999), whereas biological fouling is due to bacteria (Lepore and Ahlert 1988; Vaugham 1989; Farooque et al. 1999; Wolfaardt and Cloete 1992; Brayers and Chraklis 1981; Ridgway 1989; Saad 1992). Degradation of the membranes can be either due to physical deformation or chemical changes (hydrolysis or oxidation). Fouling and degradation of membrane not only reduce the production and quality but also result in increase in operating expenditure. Thus, it is imperative to investigate and understand the causes of any decline in RO system performance and thereby employ necessary measures to avoid either degradation or fouling of membrane. One of the causes of the decline in productivity of RO system is the chemical degradation of RO membrane itself.

Feed water for RO system is generally injected with chlorine as a biocide which can damage the membrane thereby decreasing the salt rejection and increase the permeate rate. Reverse osmosis desalination plant in Jeddah (Phase I) has been in operation since 1989, and it was discovered that due to chlorine attack, quality of permeate deteriorated (David 1991; Green et al. 2011). Chlorinated sea water also contains trace metals (Fujiwara et al. 1994; Kumano et al. 1994), which may either accelerate or retard the degradation of membrane due to their presence with residual chlorine. The presence of trace metals in water may yield a variety of oxidizing and/or reducing species, which may also be responsible for oxidation of membrane. Sea water has both oxidizing and reducing agents in solution (Nagai et al. 1994), which may react and chemically damage the membrane. It was observed that in the first two years performance was satisfactory; however, after two years performance started deteriorating slowly. Recently, it has been found that the variation of transport properties of synthetic membranes in the presence of organic substances is not due to transport coupling but is rather the result of deswelling effects caused by the interaction of the organic substances with at least the active layer of the membrane (Pusch et al. 1989; Pusch 1990). These swelling effects lead to irreversible changes of membrane properties. In another investigation, it was found that fouling is especially determined by the structure of the membrane surface (Pusch et al. 1989). From the above literature review, it is observed that the fouling and/or degradation of the membrane is determined by the structure of the membrane. To understand the structural and surface properties of the membrane, ATR/FTIR study can be used for getting in depth knowledge.

The entire field of polymer studies by FTIR spectrometry is extremely broad. Extensive reviews of Infrared polymer

spectrometry are available (Koenig 1980a, b; D'Esposito and Koenig 1978; Zahid 1993; Griffiths and Haseth 1986; Belfer et al. 1999). Vibrational spectrometry has been applied to the identification of polymeric materials for both qualitative and quantitative determination of chemical composition. Many parameters can be investigated including polymer end groups, chain branching, configuration and steric and geometric isomerism. It can also be used to identify and determine the concentration of impurities, additives and residual monomers in polymeric materials. The effects of external conditions on polymers, the change in state with temperature and pressure as well as the effects of deformation, fatiguing and weathering can also be studied. The analysis of polymers using FTIR spectrometry is done by isolation of spectral features. Spectral subtraction is frequently employed to isolate spectral features of a component in a polymer blend, to remove solvent bands or to isolate spectral features after a chemical reaction or physical change in the sample (Lin et al. 2001). By this method, known as scaled absorbance subtraction, bands of constituents of interest can be isolated and contaminants can be identified. Some studies on the effect of interactions of water and foulants with membrane surfaces by ATR/FTIR have been reported (Cho et al. 1998; Santos and Duarte 1998; MacCarthy and Rice 1985). Recent studies on the characterization on UF membranes used as pretreatment for seawater desalination have been reported using ATR/FTIR as tool (Shona et al. 2009).

But very little literature is available on the characterization of the membrane and effect of foulants on the structure that result in the degradation of the membrane. This paper describes the investigations carried out in detail the structural and surface properties of membranes using ATR/FTIR and for characterization of commercial RO membranes. Also the study presents a comparative study of new and fouled membrane to identify the adsorbed foulants under the following objectives.

1. To identify the functional groups and to understand the structural configuration of virgin (new) RO membranes.
2. To develop a methodology for measuring membrane degradation by ATR/FTIR.
3. To identify adsorbed foulants and observe their effects on the membrane structure.

## Experimental

### Membranes

Three spiral wound SWRO virgin membranes from different companies Hydranautics (type-SWC4+), Saehan (type RE 8040-SR) and Koch (KPN 8282203) were obtained. Similar membranes were installed at the Al-Jubail seawater reverse

osmosis (SWRO) plant. Membranes received pretreated sea water from Al-Jubail SWRO plant pretreatment section. The pretreatment involved the following:

Before dual media filters, ferric chloride was dosed at a concentration of 3.0 mg/l and also sulphuric acid was dosed to maintain a pH value of 6.9 at the inlet of dual media filter. Acid was further dosed just before the cartridge filters to achieve a pH value of 6.8. After the cartridge filters, sodium bisulphite (SBS) was also dosed to remove chlorine from RO feed water. As a dual protection, SBS was also dosed at the suction of high-pressure pumps. Product water was passivated by dosing limewater and carbon dioxide to maintain a positive LSI and was disinfected with calcium hypochlorite (Table 1).

### ATR/FTIR spectroscopy

ATR/FTIR analyses were performed on freeze-dried fouled membrane samples and virgin membranes of about 45 by 10 mm. IR spectra were recorded on a Perkin-Elmer spectrometer (Spectrum one) using UATR accessory. The UATR accessory contained a ZnSe/diamond flat plate crystal at a nominal incident angle of 45°, and an average depth penetration of 2 µm. Each spectrum presented is the result of 32 accumulations obtained with a resolution of 4 cm<sup>-1</sup> with air as the background. All the samples were pressed with the same force to obtain equivalent intimate contact between ATR crystal and sample surface. Spectra were corrected to take into account the wavelength dependence of ATR and baseline corrected. Difference spectra of fouled membrane from virgin membrane were obtained by subtraction method using Perkin-Elmer software. The use of spectral subtraction software supplied with FTIR unit allows the ATR spectrum of the clean membrane to be subtracted from that of the fouled membrane leaving only the ATR spectrum of the foulants.

The deposits on the membrane were removed by scraping from the membrane and freeze-dried, and the dried powder was analysed by ATR/FTIR.

### Material from membrane swatches

Membrane swatches recovered from the feed and brine end of the RO membranes were divided into strips of known surface area and the biofilm material scrapped off and organic analysis conducted by modified methyl tertiary-butyl ether (MTBE) extraction (Green et al. 2017). Mass recovered was in all the cases normalized with respect to membrane surface area.

## Results and discussion

### ATR/FTIR results: virgin membranes

A number of problems were encountered in the characterization of RO membranes. These membranes have asymmetric structure generally consisting of a thin selective skin layer supported by porous substructure (Belfer et al. 2000). ATR/FTIR spectra of virgin membranes are shown in Figs. 1, 2, 3 and 4. All the membranes show almost the same spectral pattern with slightly different intensities. The FTIR spectra are very similar to that reported in the literature for membranes made of Udel polysulphone polymer (Pihlajamäki et al. 1998; Hummel 1984). The three large peaks at 3340, 2920 and 1660 cm<sup>-1</sup> represent either aliphatic alcohols or ether alcohols (Hummel 1978). The peaks represent primary, secondary, tertiary and cyclic alcohols, and probably come from the manufacturer's preservation agent, glycerine. The peaks between wavelengths of 1700–1500 cm<sup>-1</sup> are typical of those for a polymer made of Udel polysulphone. The only peak that is unusual for polysulphone occurs at 1040 cm<sup>-1</sup>. The search programme indicates that this peak comes from an aliphatic alcohol, probably glycerine.

The intensity of peaks for the Saehan polysulphone membrane is much greater than both Koch and Hydranautics membrane. Figure 4 shows ATR/FTIR spectra of all the three virgin membranes.

**Table 1** Important plant operation parameters

#	SWRO membrane supplier	Hydranautics	Koch	Saehan
1	Model #	SWC4+	KPN 8282203	RE 8040-SR
2	Max. flux (m <sup>3</sup> /d)	24.6	19	24.6
3	Salt rejection (%)	99.8	99.8 (Cl)	99.6
4	Max. operating pressure (bar)	82.7	82.7	82.7
5	Max. operating temperature (°C)	45	45	45
6	Max. chlorine conc. (ppm)	<0.1	<0.1	<0.1
7	Max. feed water SDI	5.0	5.0	5.0

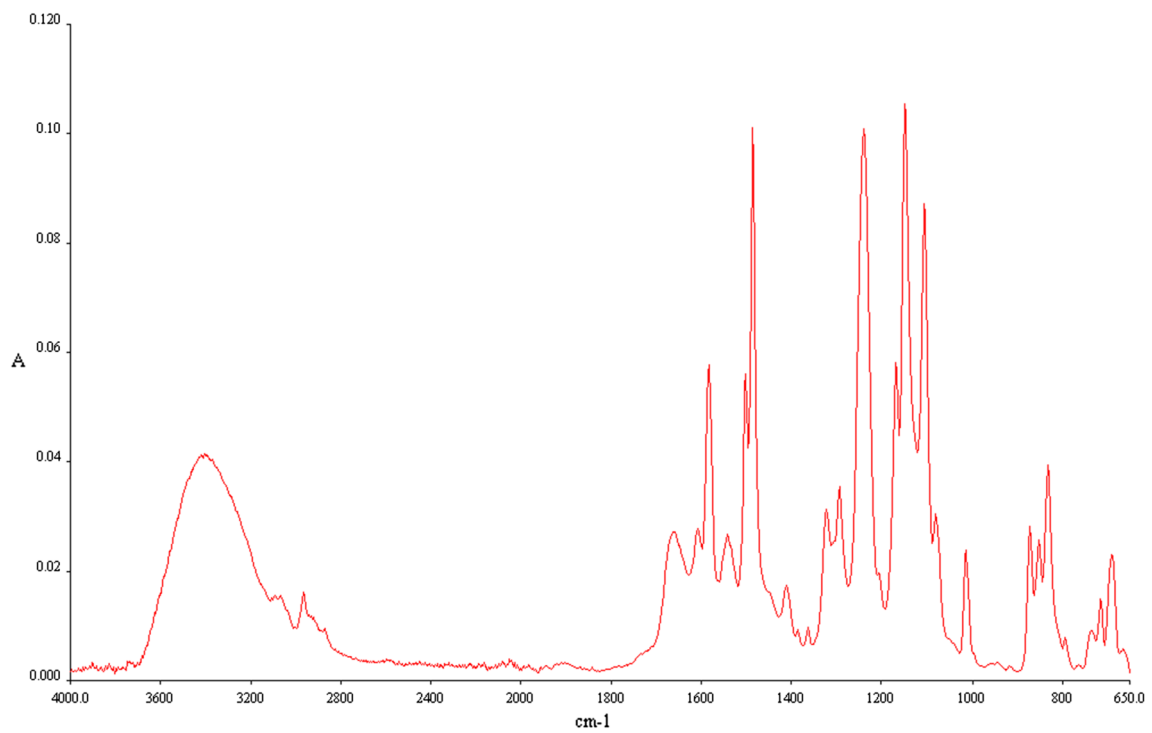


Fig. 1 FTIR spectra of the virgin Koch membrane

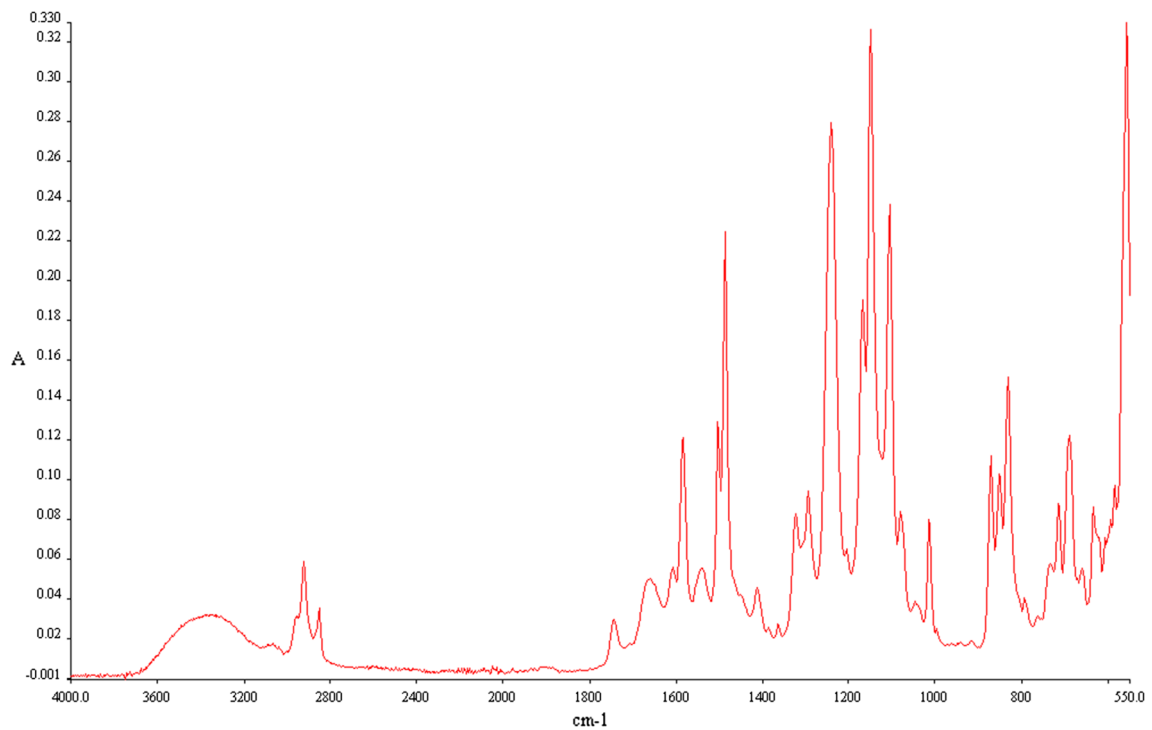


Fig. 2 FTIR spectra of the virgin Saehan membrane

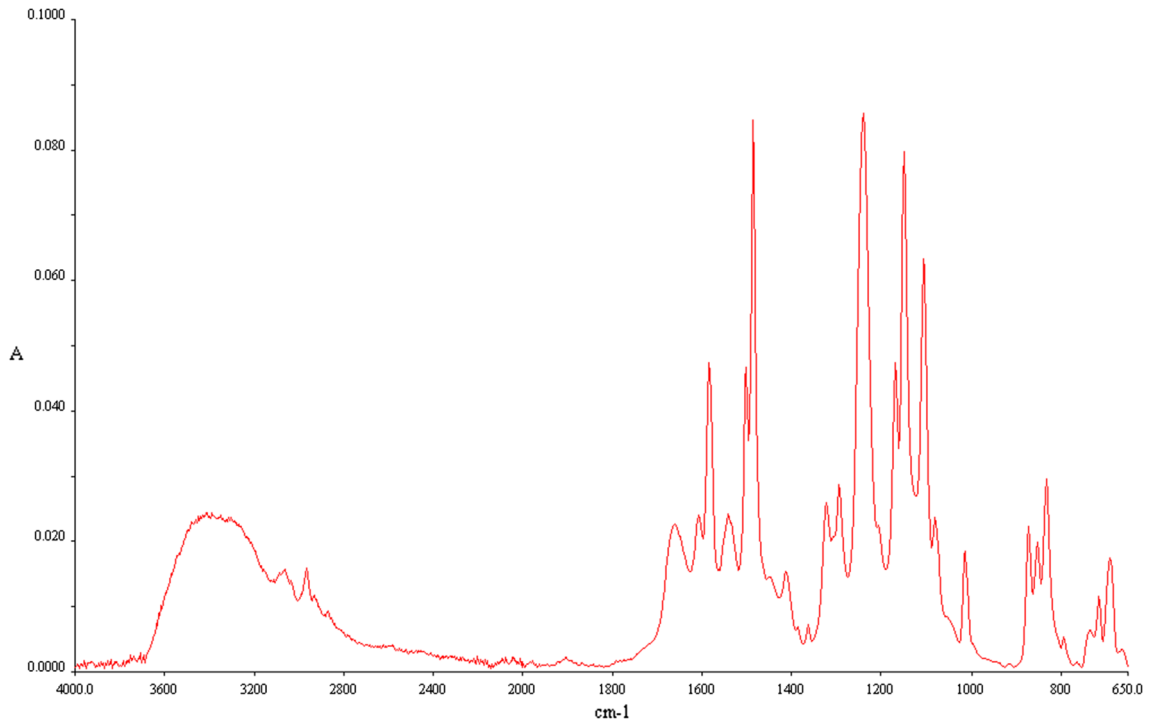


Fig. 3 FTIR spectra of the virgin Hydranautics membrane

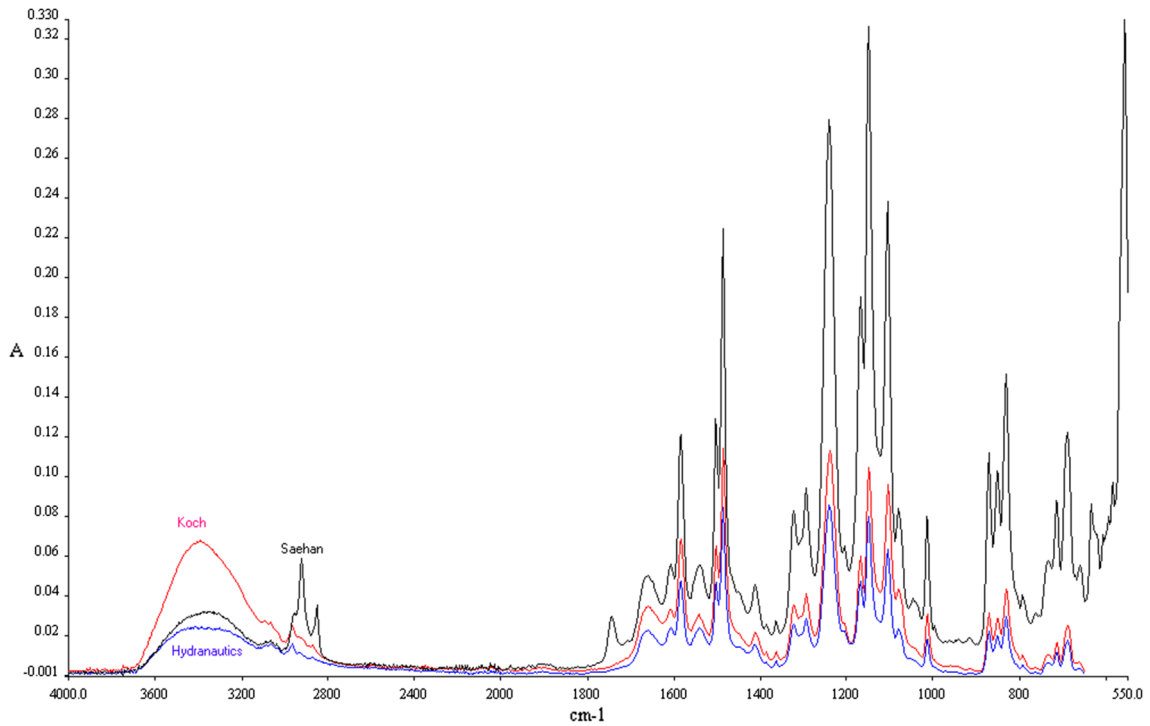
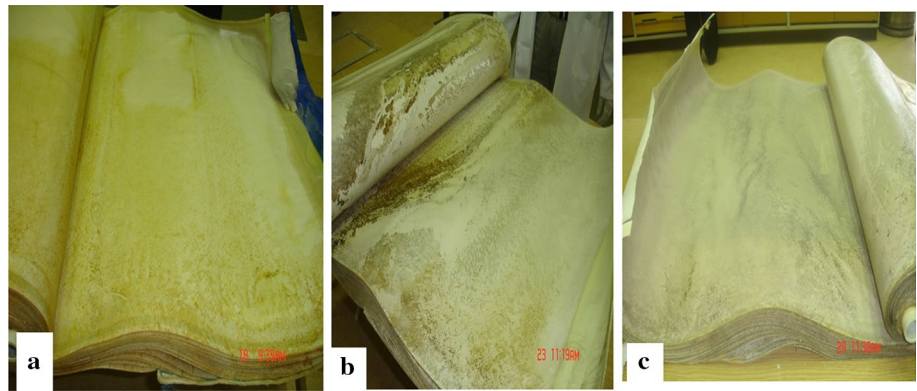


Fig. 4 FTIR spectra of the three virgin membranes

**Fig. 5** Autopsied showing fouled membrane sheets **a** Koch, **b** Saehan, **c** Hydranautics



### Visual inspection of the fouled membranes

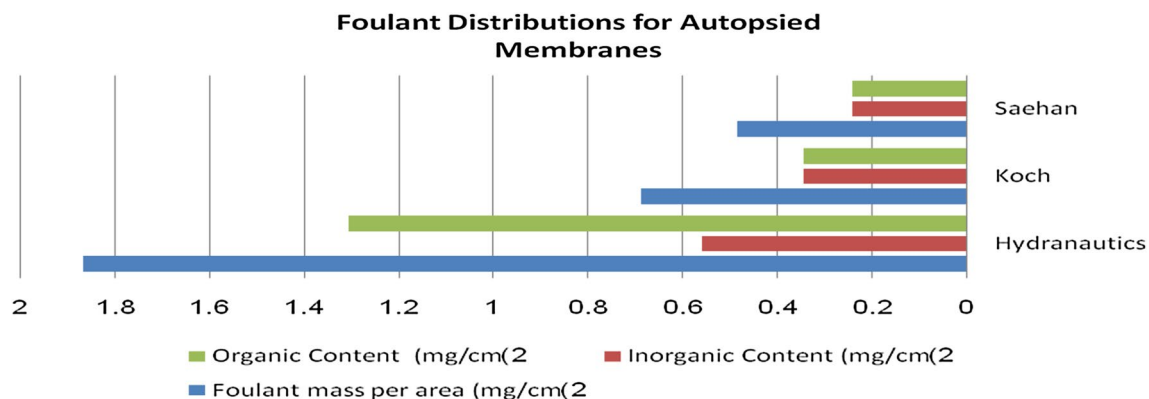
Visual inspection of membranes surface indicated that the feed surfaces were more heavily fouled than the reject element (Fig. 5). There was a general increase in density of surface foulant towards the feed end of the module. In addition to surface fouling, a brown colouration was noted on both the feed and brine end of the module. Hydranautics membrane indicated a substantial fouling on the membrane and was slightly more fouled than Saehan but less than Koch membrane. However, the membrane autopsy assay revealed that the foulant and its organic content are much higher than that of Saehan and Koch membranes (Fig. 6).

### ATR/FTIR results for fouled membranes

Figure 7 shows ATR/FTIR spectra of virgin and fouled Hydranautics membrane. There is a distinct enhancement of the peaks in the range of 3450–3200 and 3080–2850  $\text{cm}^{-1}$  indicative of membrane fouled by either hydroxy (or amino group) or phenoxy group compounds. The enhancement of the peaks at 1650  $\text{cm}^{-1}$  (amide I) and 1550  $\text{cm}^{-1}$  (amide II) is indicative of proteins. High

absorption peak at 2920  $\text{cm}^{-1}$  may be probably due to the membrane fouled by phenoxy group. The C–O stretching band associated with carbohydrate or polysaccharides is visible near 1040  $\text{cm}^{-1}$ . Figure 8 shows the difference spectrum of the fouled from the virgin Hydranautics membrane. The spectrum reveals that the foulants are composed of either hydroxy or amino groups as distinct from the bands at 3280 and 2920  $\text{cm}^{-1}$ . Also the bands at 1650 and 1550  $\text{cm}^{-1}$  may be due to combination of amides and carbonyl groups with other substituents. The peak at 875  $\text{cm}^{-1}$  is probably due to aromatic carbon. The presence of absorption in these regions suggests a significant amount of organic fouling.

As the mass of the foulants on the membrane surfaces increases, it begins to mask the IR spectrum of the membrane. The foulants vibrational bands increase in intensity relative to the membrane vibrational bands of the membrane. It is apparent that the vibrational bands for the membrane in the region 1300–1000  $\text{cm}^{-1}$  (Fig. 7) decrease in magnitude. The negative peaks near 1060  $\text{cm}^{-1}$  are due to over-subtraction of carbohydrate group. The membrane foulant(s) is a mixture of compounds, and thus, the vibrational bands are broader with overlapping bands.



**Fig. 6** Foulant distribution of autopsied membranes

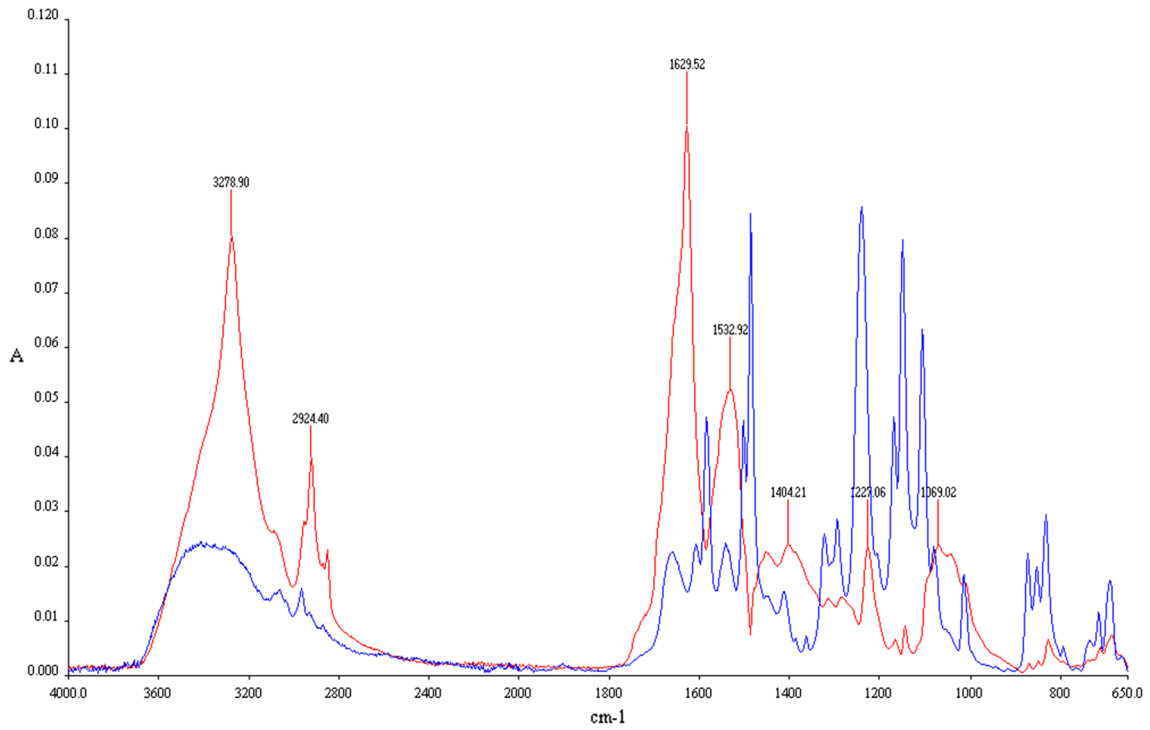


Fig. 7 FTIR spectra of the virgin (blue) and fouled (red) Hydranautics membrane

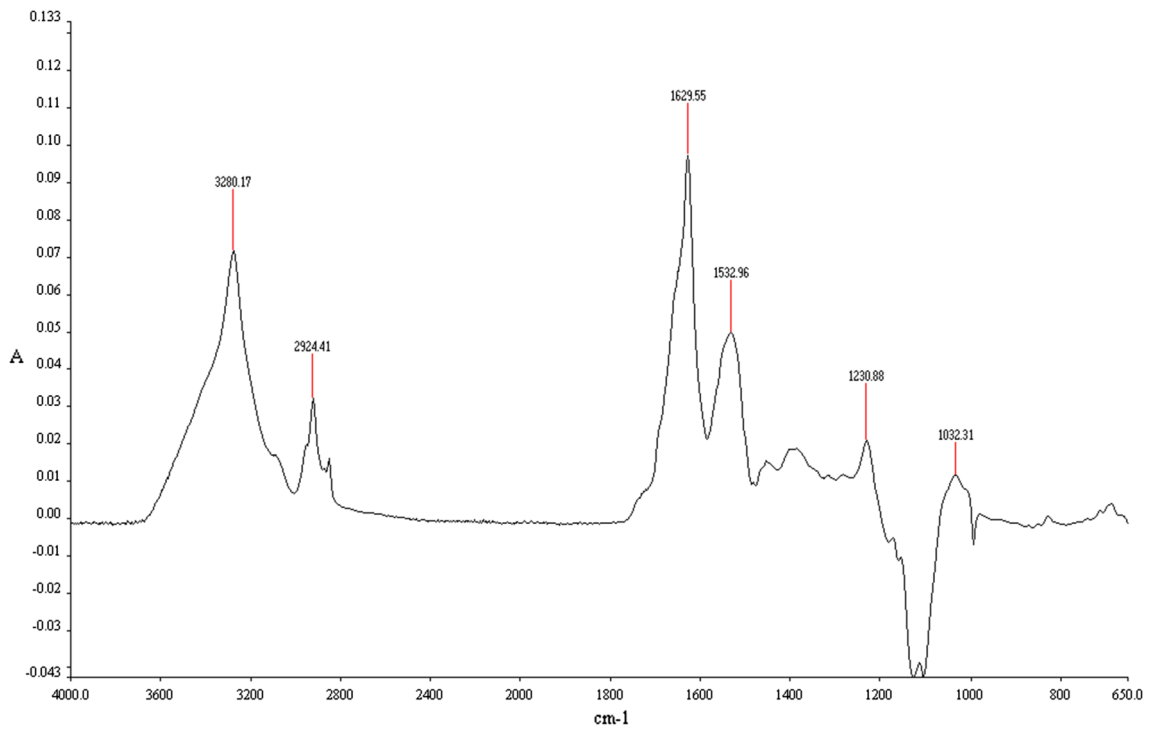
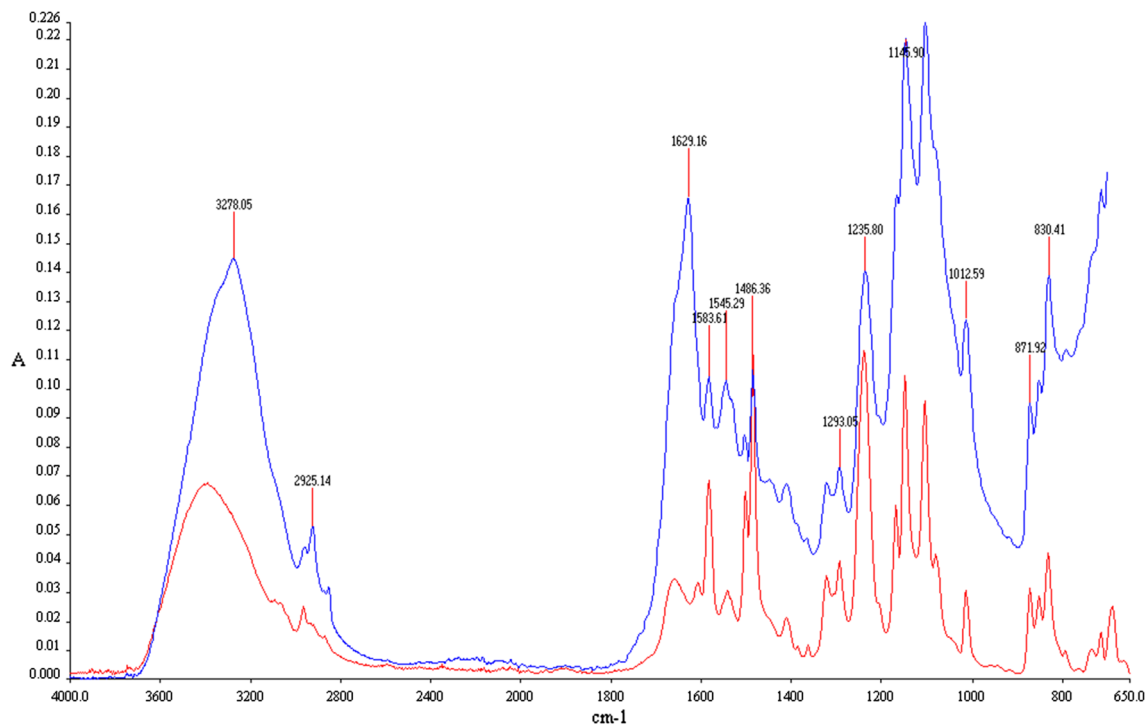
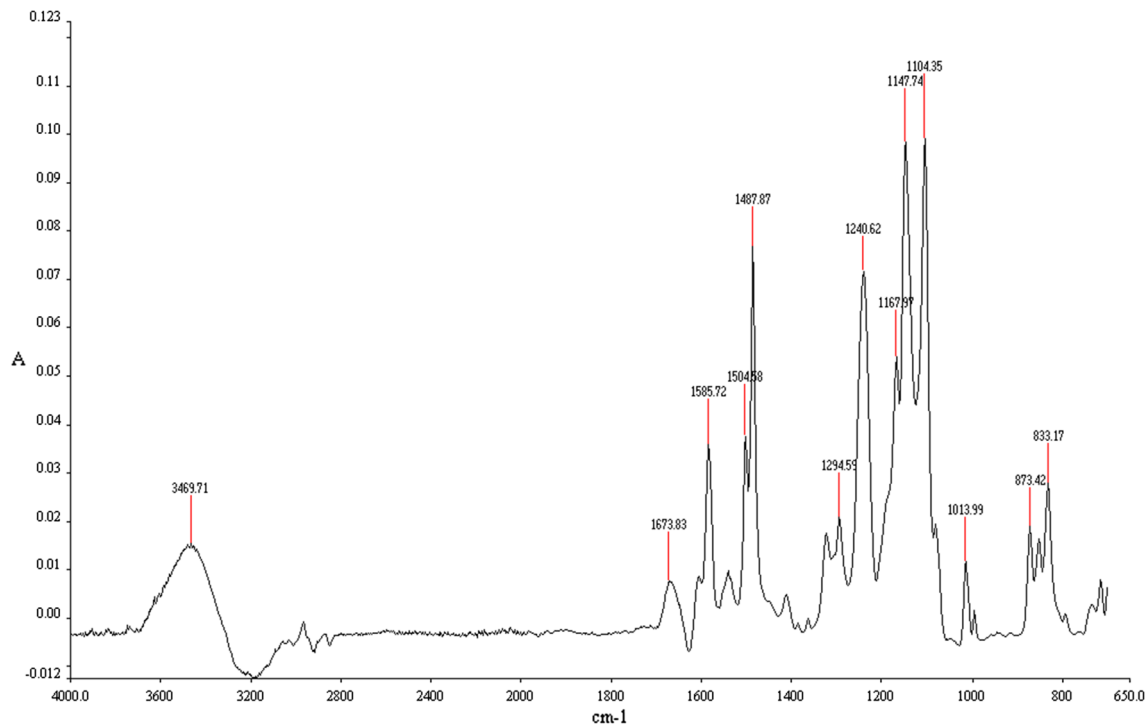


Fig. 8 Difference ATR/FTIR spectra of Hydranautics membrane



**Fig. 9** FTIR spectra of the virgin (red) and fouled (blue) Koch membrane



**Fig. 10** Difference ATR/FTIR spectra of Koch membrane

The fouled spectrum for Koch membrane in Fig. 9 shows enhancement of the peaks at 3278 and 2925  $\text{cm}^{-1}$ . This is similar to that observed for fouled Hydranautics

membrane showing fouling due to amino group, phenol or aryl amine. Also peaks in the region 1600–1100  $\text{cm}^{-1}$  show a distinct enhancement, indicating the membrane is



fouled with amide and carbonyl groups. The two negative peaks in the difference spectrum (Fig. 10) at 3200 and 1650  $\text{cm}^{-1}$  are due to over-subtraction of C-H stretching

bands. There is evidence of protein on the surface of the membrane as amide I and amide II bands are visible near 1650 and 1550  $\text{cm}^{-1}$ . Carbohydrate is also likely to be

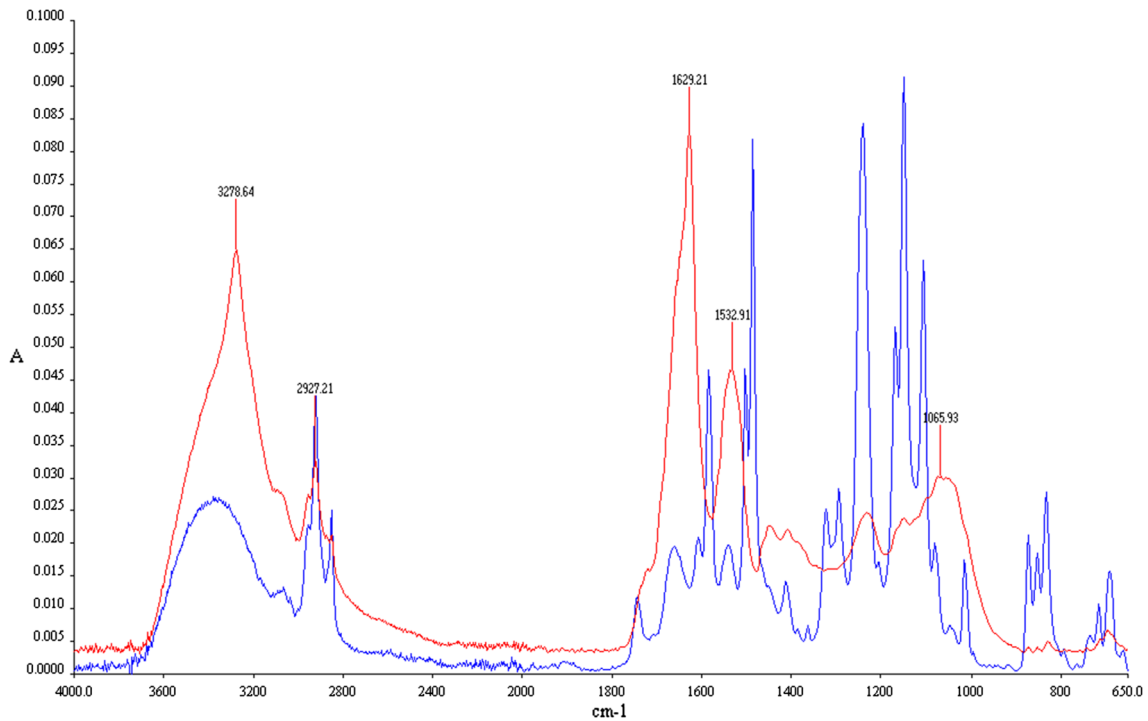


Fig. 11 FTIR spectra of the virgin (blue) and fouled (red) Saehan membrane

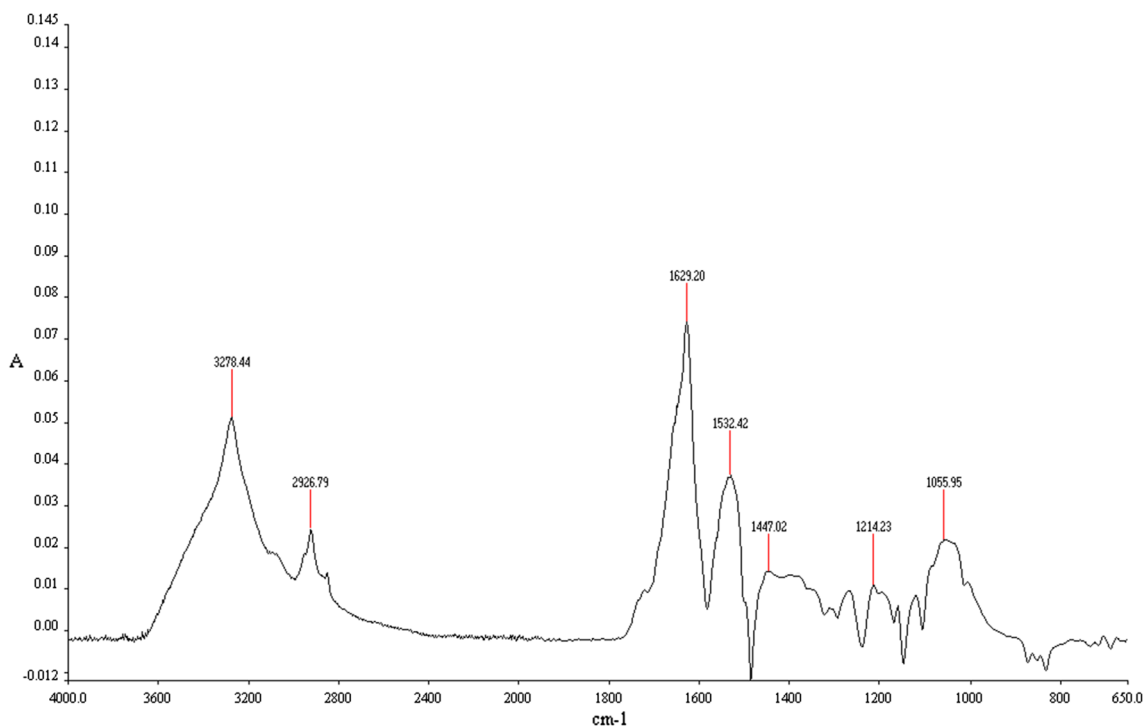


Fig. 12 Difference ATR/FTIR spectra of Saehan membrane

present as evident from the spectrum showing a peak near  $1060\text{ cm}^{-1}$ .

The fouled Saehan membrane spectrum (Fig. 11) shows enhancement of the absorption bands near  $3200$ ,  $1650$  and  $1550\text{ cm}^{-1}$  indicative of either aliphatic secondary amide or amino group, and the C–O stretching band associated with polysaccharides is visible near  $1060\text{ cm}^{-1}$ . This is evident from the difference spectrum (Fig. 12) which shows distinct bands in this region. The vibrational bands for the membrane in the region  $1300$ – $1000\text{ cm}^{-1}$  show significant decrease in magnitude indicating fouling of the membrane. Some negative peaks observed in the difference spectrum at  $1500$ ,  $1240$  and  $1150\text{ cm}^{-1}$  are probably due to over-subtraction.

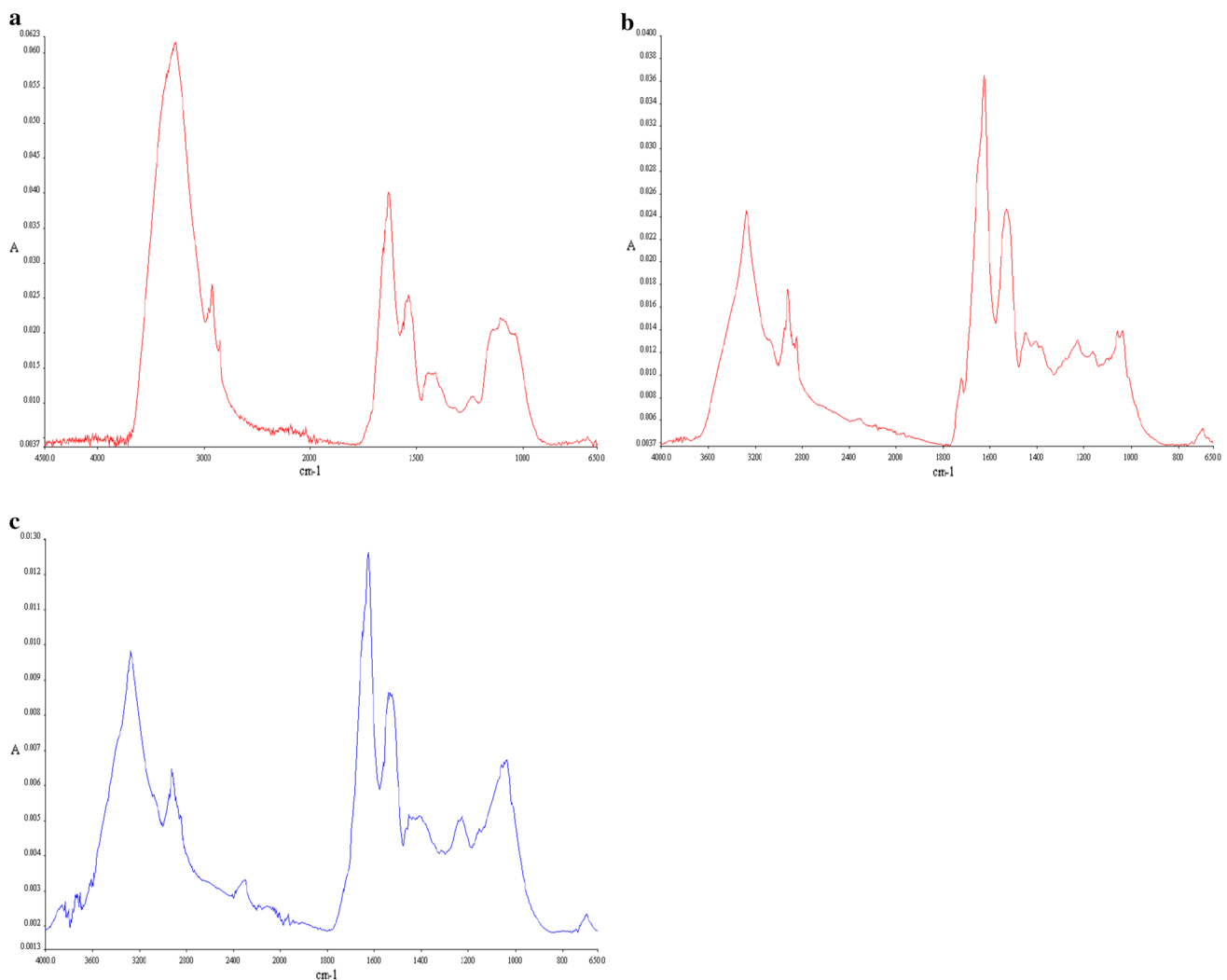
### ATR/FTIR of foulant deposits

ATR/FTIR analysis was conducted to investigate the functional groups in the foulant deposits on the membrane surface

(Fig. 13). Spectra of foulant deposits for all three membranes showed the bands in the range of  $3400$ – $3150\text{ cm}^{-1}$  indicating either amino or aliphatic secondary amide groups and near  $1650$  and  $1550\text{ cm}^{-1}$  indicating amide group.

### Conclusions

1. ATR/FTIR spectra of all the three virgin membranes showed almost the same spectral pattern with slightly different intensities. The FTIR spectra are very similar to that reported in the literature for membranes made of Udel polysulphone polymer.
2. Fouled membranes showed a distinct enhancement of the peaks in the range of  $3450$ – $3200$  and  $3080$ – $2850\text{ cm}^{-1}$  indicating the membranes fouled by either hydroxy (or amino group) or phenoxy group compounds.



**Fig. 13** ATR/FTIR spectra of Foulant deposits on membranes **a** Koch, **b** Hydranautics, **c** Saehan

3. ATR/FTIR indicated a distinct enhancement to the amide I ( $1650\text{ cm}^{-1}$ ) and amide II ( $1550\text{ cm}^{-1}$ ) bands indicative of protein, and the C–O stretching band associated with polysaccharides is visible near  $1040\text{ cm}^{-1}$ .
4. The ATR/FTIR difference spectra and of foulant deposits spectra confirmed that the RO membrane surfaces were mostly proteins and carbohydrates or polysaccharides.

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