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RSM optimized soy protein fibre as a sorbent material for treatment of water contaminated with petroleum products

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ABSTRACT

The aim of this study is to investigate the use of modified soy protein fibre (SPF) as a sorbent material for treatment of water contaminated with petroleum products. Studies were done with diesel, petrol, kerosene and petroleum ether both in pure oil medium and oil–water medium. Oleophilic behaviour of SPF was enhanced by successive benzoylation (Bz-SPF) and graft copolymerization. Response surface methodology was applied to find maximum optimum graft percentage as per IV optimal design comprising of 31 experimental runs. Process variables investigated were monomer concentration, reaction temperature, reaction time, potassium persulphate (KPS): Ferrous ammonium sulphate (FAS) molar ratio and pH. Percentage graft yield was monitored as response and characterized through FTIR and SEM techniques. There is antagonist two-factor interaction between monomer × KPS: FAS ratio with significant negative main effect of KPS:FAS molar ratio on graft yield. Chemical modification of SPF, enhanced water resistance and acid–base resistance properties. Bz-SPF and Bz-GSPF showed high sorption capacities for diesel (30.7–43.55 g/g), petrol (23.6–37.72 g/g), kerosene (15.5–34.06 g/g) and petroleum ether (9.7–28.88 g/g) both in pure oil medium and oil–water medium. Thus, eco-friendly SPF shows high performance as sorbent material for removal of oil.

Keywords: Biopolymer; Benzoylation; Design of experiments (DOE); Response surface methodology (RSM); Graft yield

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1. Introduction

Oils in different forms like crude oil, diesel, kerosene and gasoline contaminate water. Oily waste from industries metal manufacturing, food processors and petroleum refining releases during various stages of production, transportation, refining and use causes serious environmental problems [1,2]. Many technologies like chemical treatment, cyclone separation, cartridge treatment, microfiltration and ultrafiltration

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have been developed and used for treatment of water contaminated with oil [3–8]. Among different technologies developed, use of sorbents is most common technique to treat oily water due to possibility of collection and reuse. Sorbents used may be synthetic polymers or natural materials. Polypropylene, polyethylene and polyurethanes are commonly used synthetic polymers [9–11]. These are quite efficient but major drawback is their non-biodegradability. Natural polymers like chitosan, cotton, milkweed, kenaf, wool, silkworm cocoon waste, rice straw and vegetable fibres have been studied as materials for oil removal [12–17]. Natural polymers and their derivatives showed high oil-sorption capacities. Moreover, these materials have advantage of being abundant availability, biodegradability and environmental friendly nature.

Soybean, once a relatively minor farm commodity, now becomes a substantial cash crop and world's most abundant source of vegetable protein. As a result of chemurgic movement of 1930s, soy protein has been developed for different industrial uses like plastics, adhesives, textile fibre, paper coating and fire-fighting foams [18]. Commercially, soy is available in different grades: Soy flour, soy protein concentrate and soy protein isolate, depending upon the amount of protein present. Soy protein fibre (SPF) is a man-made regenerated protein fibre from soybean protein which is blended with pol(vinyl alcohol). Regenerated SPF is a cost-effective competitive production material for fibres in textile industry, abundant with higher protein content (40%), compared to peanuts (25%) and corn (10%) with environmentally friendly production. Soybean fibres are cream-coloured fibre with their diameter around 20 μm. It contains 18 amino acids including polar functional groups like carboxyl, amine and hydroxyl [19]. The presence of reactive functional groups provide site for the chemical modifications which improve the functional properties of proteins. Acylation, cross-linking, oxidation, blending with other polymers and copolymerization are some techniques to modify the structure of proteins to enhance or modify the functional and physiochemical properties [20–22]. Graft copolymerization is one of the promising techniques to improve the physical and chemical properties of biopolymers. Various workers have carried out graft copolymerization of vinyl monomers onto proteins using chemical and radiation techniques to incorporate desired properties [23–26].

The aim of this study was to develop biopolymer-based material as sorbent to remove different oils from aqueous medium so that it can be utilized for treatment of water contaminated with different petroleum products. Diesel, petrol, kerocene and petroleum ether have been taken as model oils for this study. The hydrophobic behaviour was incorporated onto SPF through benzoylation and graft copolymerization with methylmethacrylate. Graft percentage was improved through design of experiments using response surface methodologies approach. Modified soy protein fibre (Bz-GSPF) with maximum graft yield was studied for acid–base resistance and water resistance behaviour. The modified fibre synthesized under optimum conditions was analyzed for oil sorption from different oils in aqueous medium and in pure oil medium.

2. Experimental

2.1. Materials

SPF was purchased from Swicofil Ag Textile services, China. Methyl methacrylate (MMA) was obtained from Merck chemicals. Ferrous ammonium sulphate, potassium persulphate, sodium hydroxide pellets and hydrochloric acid were procured from SD Fine chemicals Ltd., Mumbai. Solvents used were of analytical grade. Samples were weighed on Afcoset make electronic balance (ER-200 A).

2.2. Benzoylation of SPF

Benzoylation of SPF was carried out using 5.0 g of fibre, which was immersed in solution of 100 ml of 10% sodium hydroxide. This was followed by the addition of 10 g of benzoyl chloride to the reaction flask. The reaction flask was stoppered and mixture was shaken vigorously. The stopper was removed time to time to release CO_2 pressure. After the disappearance of odour of benzoyl chloride, the mixture was acidified with dilute hydrochloric acid and filtered. Thereafter, fibre was extracted with cold ether to remove traces of benzoic acid. Benzoylated soy protein fibre (Bz-SPF) was dried in oven at 50°C till a constant weight was obtained [27].

2.3. Graft copolymerization of MMA onto SPF

A known weight of SPF (500 mg) was taken in a flask containing distilled water. A definite molar ratio of KPS-FAS was added to the reaction flask and a known amount of monomer was added drop by drop with constant stirring. The reaction was performed under a definite temperature for specific time. Homopolymer formed was extracted with acetone for 24 h and grafted Bz-GSPF thus obtained was dried at 40°C until constant weight was achieved. Optimum conditions of reaction time, pressure, solvent amount, initiator ratio, pH and monomer concentration were worked-out to get maximum graft percentage (P_g). Percentage graft yield was calculated as per Eq. (1) [28]:

$$P_g = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

where P_g = percentage graft yield; W_1 = initial weight of sample; W_2 = final weight of sample (after removal of homo polymer).

2.4. Response surface optimal design

Response surface optimal design was selected for optimization of graft copolymerization. There are total of 31 experimental runs as per IV optimal criteria, which include 21 model points, five points to test lack of fit and five replicates. This is an algorithm-based design with emphasis on lower average prediction variance across the design space. Five independent variables tested were monomer concentration between 4.22 and 5.63 mmol/L, reaction temperature between 70 and 90°C, reaction time between 200 and 260 min, KPS(1):FAS(x) molar ratio between 1.55 and 2.0 and pH between 7.5 and 9.0. The ranges of these variables were selected through one-factor-at-a-time approach along with trial experimentation. Graft yield (P_g) was taken as the response variable. The ANOVA modelling was performed and significant process variables were selected based on *p*-value. Model selection was based on best model fitting, non-significant lack of fit and normal distribution of predicted variables along with prediction capabilities (R^2) statics.

2.5. Characterization

Grafted and ungrafted SPF were characterized by FTIR spectra recorded with the help of Perkin Elmer FTIR spectrophotometer using KBr pellets. Scanning electron microscopic studies of SPF, Bz-SPF and Bz-GSPF were carried out on electron microscope machine (JEOL JSM 6610LV). A thin conductive layer was sputtered to make the samples conducting to avoid charging.

2.6. Physicochemical properties

Various physicochemical properties such as water uptake studies, acid and base resistance were studied for grafted vis-a-vis ungrafted SPF. For water uptake studies, a known weight of each ungrafted fibre (SPF), benzoylated fibre (Bz-SPF) and grafted fibre (Bz-GSPF) sample (0.1 g) were immersed in 50 ml of distilled water and weight of each sample was taken after every 3 h interval till a constant weight was obtained. The percentage water uptake by each sample was calculated.

$$P_w = \frac{W_f - W_i}{W_i} \times 100 \quad (2)$$

where P_w = percentage water uptake; W_i is initial weight of sample; W_f is the final weight of sample.

Acid and base resistance studies were carried out by putting a known weight of samples (0.1 g) in 25 ml of 1 N HCl and 1 N NaOH. The weight of each sample was noted down after every 6 h interval till a constant value was obtained. Percentage weight loss was calculated as:

$$P_{wl} = \frac{W_{iw} - W_{fw}}{W_{iw}} \times 100 \quad (3)$$

where P_{wl} = percentage weight loss; W_{iw} is initial weight of sample and W_{fw} is the final weight of sample.

2.7. Oil sorption studies

Oils employed to investigate the sorption on sorbents were petrol, diesel, kerosene and petroleum ether. The viscosities were measured using a redwood viscometer. Sorption of these oils on different sorbents: SPF, Bz-SPF and Bz-GSPF was determined. Sorption studies were done in pure oil medium and in the presence of water.

2.7.1. Sorption of oil in pure medium

To analyse sorption capacity of fibre in pure oil medium without water, 50 ml of oil was placed in 250 ml beaker and 0.1 g of sorbent was immersed for 24 h. Sorption capacity of sorbent was determined by taking initial weight of sorbent and weight of wetted sorbent.

2.7.2. Sorption of oil in water bath

50 ml of water was taken in 500 ml beaker. 50 ml of oil and 0.1 g of sorbent was added to it. Sorption capacity was studied for different time intervals up to 24 h. At the end of respective time period, sorbent was taken out; extra oil was allowed to drain in beaker for 15 min and then weighed. Sorption capacity was expressed in g of oil absorbed per g of sorbent [29]. The samples were drawn at different time intervals up to equilibrium time.

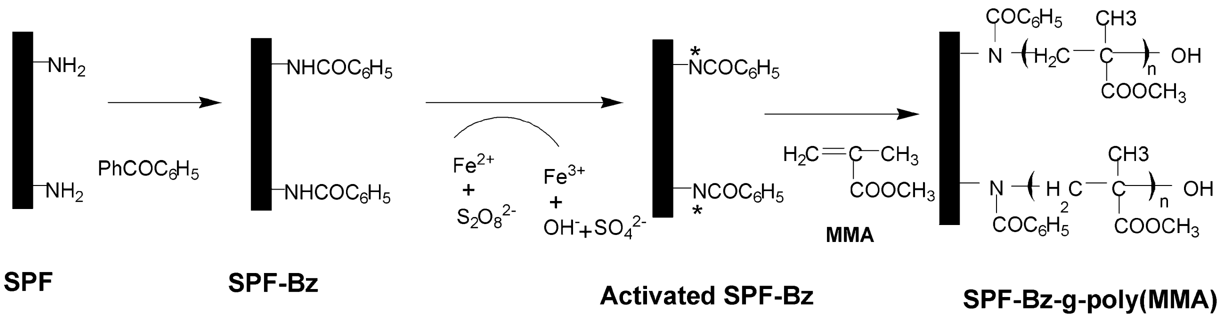

Fig. 1. Schematic representation of benzoylation and graft copolymerization of SPF.

3. Results and discussions

3.1. Screening of process variables for maximizing graft percentage

SPF was modified by benzoylation and successive graft copolymerization. Schematic representation of reaction was given in Fig. 1. RSM optimal design along with % graft yield as response variable is given in Table 1. The highest graft percentage (392%) was achieved at 5.63 mmol/L monomer concentration, 88˚C reaction temperature, 247 min reaction time, 1.67 as KPS(1):FAS(x) ratio and pH of 9. The lowest graft yield of 7% was obtained at 4.22 mmol/L monomer concentration, 70˚C reaction temperature, 200 min reaction time, 2.5 as KPS(1):FAS(x) molar ratio and pH of 7.5. The experimental data were subjected to ANOVA and the fitted model was significant at 99.99% but model lack of fit was also significant, which indicates a weak signal as compared to model noise. The Box–Cox plot recommended square root transformation (Fig. 2). Experimental data were then subjected to ANOVA modelling and the sequential model sum of squares predicted that both linear and the quadratic model best fits the response data and non-significant lack of fit. As quadratic model has higher R^2, thus reduced quadratic model was built by eliminating non-significant model ($p > 0.1$) terms using the backward elimination method. The reduced quadratic model was highly significant at 99.99% ($F = 33.5$, degree of freedom = 7) and non-significant *lack of fit* ($p = 0.1654$) (Table 2). Before moving to response plotting, diagnostic statistics were analysed to check normality of the residuals and actual vs. predicted plot of graft yield (Fig. 3) to check any potential outliers. The fitted model explained 91% variance with coefficient of variation (CV) of 15.70% and signal to noise ratio of 15.5 (>4 is recommended). Thus, the model can be navigated in the design space. The unit less regression coefficients are given in Eq. (4) and in actual process factors in Eq. (5).

$$\begin{aligned}\text{Sqrt (Pg)} = {} & 14.74 + 0.35\ \text{A} + 0.46\ \text{C} - 5.60\ \text{D} \\ & - 1.10\ \text{A x D} - 2.41\ \text{A}^2 + 2.40\ \text{C}^2 - 3.11\ \text{D}^2\end{aligned} \tag{4}$$

where P_g = percentage graft yield; A = monomer concentration, C = reaction time and D = KPS(1):FAS(x) are in coded units.

$$\begin{aligned}\text{Sqrt (Pg)} = {} & -47.864 + 54.549\ \text{A} - 1.025\ \text{C} + 53.906\ \text{D} \\ & - 2.923\ \text{A x D} - 4.291\ \text{A}^2 + 2.262 \\ & \times 10^{-3}\ \text{C}^2 - 12.438\ \text{D}^2\end{aligned} \tag{5}$$

where P_g = percentage graft yield; A = monomer concentration (mmol/L), C = reaction time (min) and D = KPS(1):FAS(x) are in actual units.

There is significant two-factor interaction (2FI) between monomer concentration and KPS:FAS ratio. To maximize the P_g, KPS:FAS molar ratio should be set at minimum value and monomer concentration should be fixed around 5.17 mmol/L (Fig. 4). The other optimized process condition is 29 min reaction time. The reaction temperature and pH are not included in ANOVA modelling, but these parameters should be set around 90˚C and pH 9 for best results. Perturbation plot indicated the response sensitivity of each process variable and a steep slope indicated about higher change in graft yield as compared to flat slopes. The KPS:FAS molar ratio has most significant affect on percentage graft yield (Fig. 5).

3.2. Fourier transform infrared (FT-IR) spectroscopy

FTIR spectrum of the SPF showed broad peak at 3,300.3 cm^{-1} due to free –OH and –NH groups (Fig. 6(a)). Peaks were observed at 1,648.4 cm^{-1} due to N–H stretch (amide-I) and at 1,547.6 cm^{-1} due to C–O

Table 1
Experimental design (IV-optimal) along with observed responses for graft copolymerization

Std	Factor A Monomer conc. (mmol/L)	Factor B Reaction temp. (°C)	Factor C Reaction time (min)	Factor D KPS(1):AFS(x) ratio	Factor E pH	Response Graft Copoly (%)
1	5.63	70	200	1.50	7.5	334
2	4.22	90	200	1.50	7.5	226
3	5.63	90	260	1.50	7.5	350
4	4.22	70	260	1.62	7.5	211
5	4.22	70	200	2.50	7.5	7
6	5.63	90	200	2.50	7.5	12
7	5.63	70	260	2.50	7.5	26
8	4.22	90	260	2.50	7.5	63
9	4.64	90	235	1.82	7.9	164
10	4.64	90	235	1.82	7.9	288
11	4.43	79	200	1.95	8.0	303
12	4.43	79	200	1.95	8.0	290
13	4.90	80	260	1.50	8.2	333
14	4.90	80	260	1.50	8.2	325
15	5.63	80	230	2.00	8.3	135
16	5.63	80	230	2.00	8.3	130
17	4.22	70	226	1.50	8.4	220
18	5.42	70	240	2.50	8.5	17
19	5.63	90	200	2.23	8.6	51
20	4.22	90	200	2.50	8.9	58
21	4.22	77.8	200	1.50	9.0	218
22	5.63	90	200	1.50	9.0	295
23	5.63	70	260	1.50	9.0	385
24	4.22	90	260	1.50	9.0	256
25	5.63	87.7	247	1.67	9.0	392
26	4.85	70	200	1.95	9.0	350
27	5.63	70	200	2.50	9.0	28
28	4.92	82	229	2.50	9.0	46
29	4.92	82	229	2.50	9.0	41
30	4.22	70	260	2.50	9.0	32
31	5.63	90	260	2.50	9.0	72

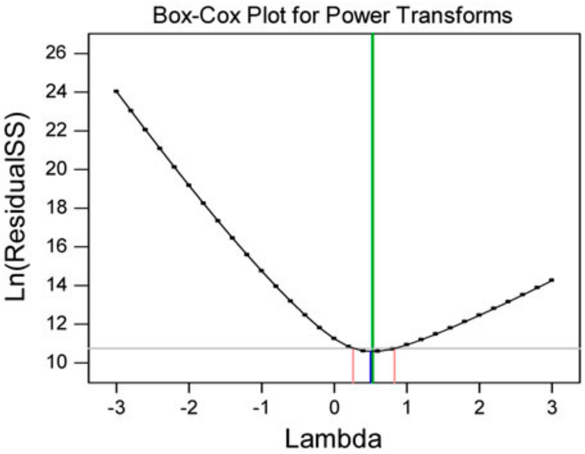

Fig. 2. Box–Cox plot showing optimized lambda for the transformation of graft copolymer.

stretch (amide II). Benzoylated SPF (Fig. 6(b)) showed peaks at 1,740.2 cm^{-1} due to C=O stretch of benzoyl group and at 1675.4, 1,518.3 and 1,462.5 cm^{-1} due to incorporation of benzene ring. Bz-GSPF (Fig. 6(c)) showed a sharp peak at 1,732.6 cm^{-1} due to >C=O group of MMA. Along with that, decrease in the intensity of amide-I (1,653.3 cm^{-1}) peak and disappearance of amide-II peak were observed. It showed that graft copolymerization occurred onto the amide group of SPF.

3.3. Scanning electron microscopy

SEM images revealed the morphological changes on graft copolymerization of MMA onto benzoylated SPF (Fig. 7). There was a clear distinction between grafted and ungrafted SPF. SPF fibre has smooth and homogeneous surfaces which get swollen on benzoylation. Graft copolymerization resulted in the

Table 2
ANOVA table for reduced quadratic model for graft copolymerization

Source	Sum of squares	Degree of freedom	Mean squares	*F*-value	*p*-value, Prob > F
Model	876	7	125.1	33.5	<0.0001[a]
A-Monomer	2.6	1	2.6	0.7	0.4148[b]
C-Reaction time	4.2	1	4.2	1.1	0.301[b]
D-KPS(1):FAS(x) ratio	669.5	1	669.5	179.2	<0.0001[a]
A × D	20.9	1	20.9	5.6	0.0268[a]
A^2	24	1	24	6.4	0.0185[a]
C^2	18.3	1	18.3	4.9	0.0372[a]
D^2	40	1	40	10.7	0.0033[a]
Lack of Fit	77.1	18	4.3	2.4	0.1654[b]
Model statistics					
Standard deviation	1.93		R^2		0.911
Mean	12.31		Adjusted R^2		0.884
CV (%)	15.70		Predicted R^2		0.842

[a] Significant at $p < 0.05$.

[b] Not-significant at $p < 0.05$.

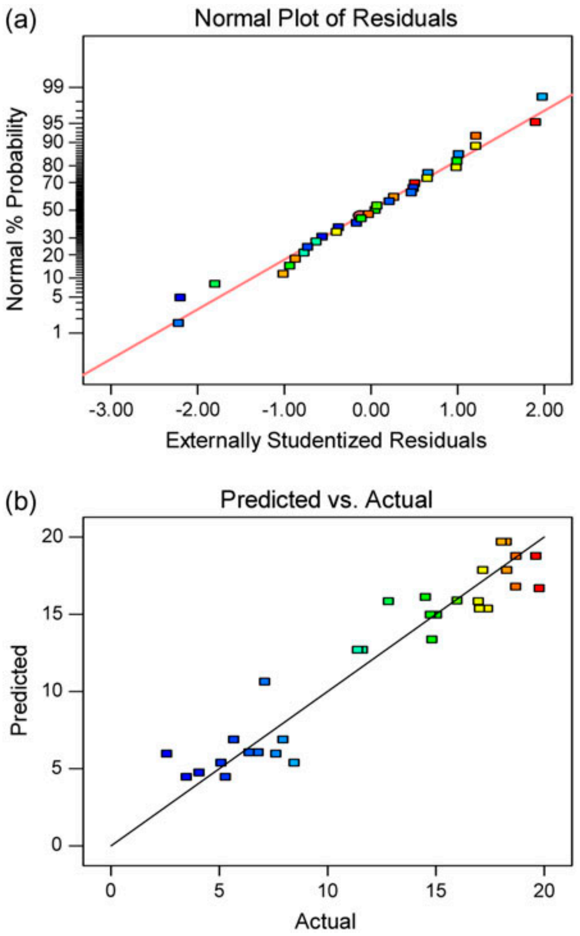

Fig. 3. (a) Normal % probability plot of graft copolymer yield for reduced quadratic model. (b) Actual vs. predicted plot of graft copolymer yield as per reduced quadratic model.

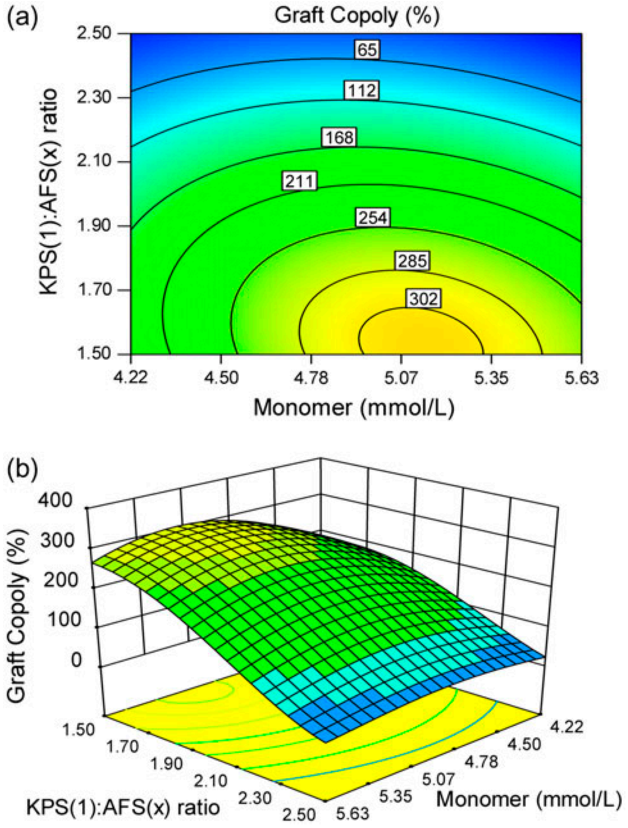

Fig. 4. (a) Two-dimensional. (b) Three-dimensional contour plot showing 2FI between monomer and KPS:AFS ratio vs. graft copolymer yield.

rough and heterogeneous surfaces due to incorporation of poly (MMA) chains [30]. The increase in roughness further prompts sorption as it enhances trapping and physical adsorption of oil on surface of sorbent.

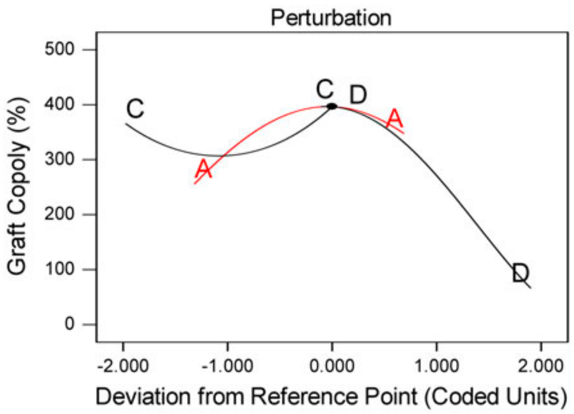

Fig. 5. Perturbation plot showing response sensitivity at optimized conditions for maximized graft copolymer yield (%). A: Monomer concentration, C: Reaction time, D: KPS (1):AFS(x) ratio.

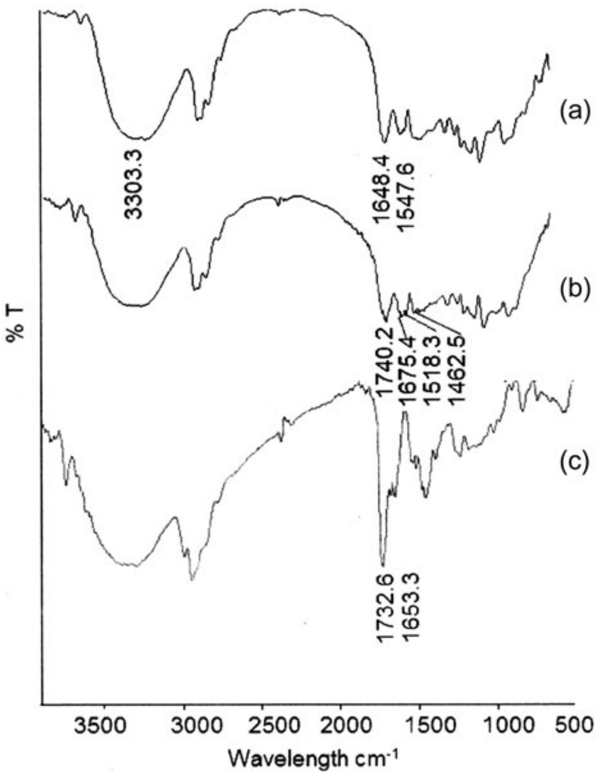

Fig. 6. FTIR of (a) SPF, (b) Bz-SPF, and (c) benzoylated grafted soy protein fibre (Bz-GSPF).

3.4. Physiochemical properties

The modified fibre was observed to be more acid and base resistant as compared to ungrafted one. SPF exhibited 80.7% weight gain in the presence of 1 N HCl, whereas Bz-SPF fibre showed 32.1% and Bz-GSPF revealed only 20.2% weight gain (Fig. 8(a)). In the presence of 1 N base, SPF showed 92.9 wt.% gain whereas benzoylated and grafted fibre showed only 43.2 and 25.7 wt.% gain, respectively (Fig. 8(b)). This was due to the fact that MMA grafted onto SPF have less affinity for 1 N HCl and 1 N NaOH as compared to ungrafted fibre. Therefore, the resistance of grafted fibre towards acid–base was found to increase with the incorporation of poly (MMA) chains on the active sites of the backbone. Water uptake studies showed that benzoylation as well as graft copolymerization resulted in the decrease in water uptake behaviour of fibre (Fig. 8(c)). SPF revealed 99.7% weight gain in 24 h whereas benzoylated SPF showed 81.3 wt.% gain. On the other hand, graft copolymerization of fibre exhibit sharp decline in water uptake with only 22.4 wt.% gain in 24 h. This was due to the fact that incorporation of poly MMA chains increased the hydrophobicity of fibre due to which water uptake decreased.

3.5. Oil-uptake studies of different oil–water emulsions

3.5.1. Pure oil medium

The study of sorbent capacity of sorbent in pure oil medium gives the maximum absorption of oil absorbed by a sorbent. The sorbent capacities of different oils are shown in Fig. 9. Maximum sorption capacity has been observed in case of diesel, followed by petrol, kerosene and petroleum ether. Results are in line with the viscosities of different oils. Sorption of oil involves two steps. Initial adsorption of oil on sorbent surface which is mainly influenced by hydrophobic interactions and then, absorption of oil into sorbent which occur by capillary action. Increase in viscosity decreases the rate of absorption, but simultaneously enhances the initial adherence of oil to sorbent surfaces as well as decreases the drainage of oil from sorbent. It results in the overall increase in the sorbent capacity with increase in the viscosity of oil. In case of different sorbents, Bz-SPF and Bz-GSPF showed higher sorption capacity as compared to untreated SPF. Bz-SPF showed sorption capacity of 30.7 g/g (diesel), 23.6 g/g (petrol), 15.5 g/g (kerosene) and 9.7 g/g (pet ether) whereas, Bz-GSPF revealed even higher sorption capacities of 42.6 g/g (diesel), 36.4 g/g (petrol), 32.6 g/g (kerosene) and 27.4 g/g (pet ether) as compared to 9.8 g/g (diesel), 7.5 g/g (petrol), 6.3 g/g (kerosene) and 4.0 g/g (pet ether) for raw SPF. The higher sorption capacity of Bz-GSPF could be due to hydrophobic nature of poly MMA chains grafted on fibre. Along with that, presence of benzoyl group also enhances the hydrophobic

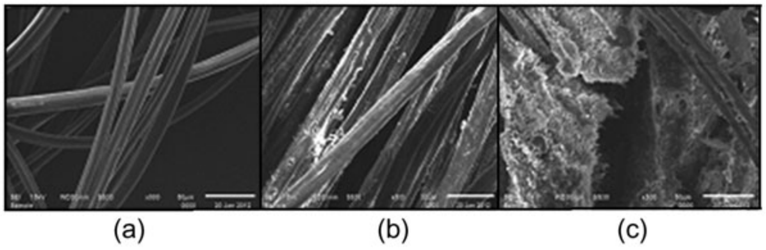

Fig. 7. SEM image of (a) SPF, (b) Bz-SPF, and (c) Bz-GSPF.

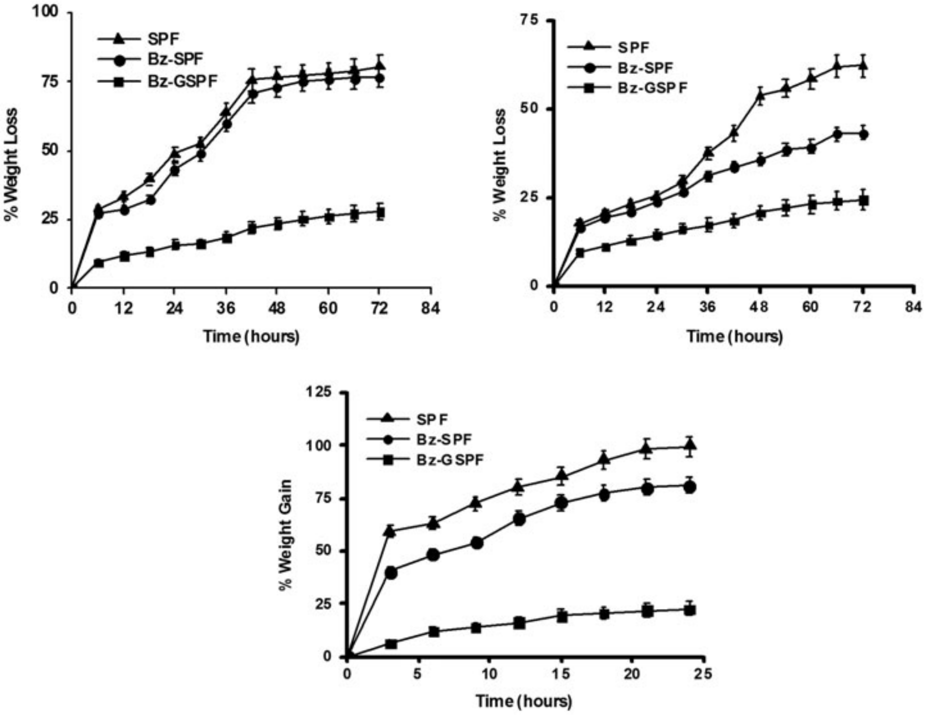

Fig. 8. (a) Acid resistance, (b) Base resistance, and (c) Water resistance studies.

character. This also explains the higher sorption capacity of Bz-SPF as compared to SPF.

3.5.2 Sorption in oil–water medium

To analyse sorption capacities of Bz-SPF and Bz-GSPF in wet environment, sorption studies were done for different oils in oil–water medium. Sorption capacity is higher in pure oil medium than in oil–water medium, as presence of water interferes with the initial accumulation of oil to fibre surface. However, both Bz-SPF and Bz-GSPF still showed higher sorption capacities as compared to SPF fibre. In the presence of water, oil sorption capacities of Bz-SPF for diesel, petrol, kerosene and petroleum ether were 31.54, 24.56, 16.62 and 10.81 g/g whereas, Bz-GSPF showed 43.55, 37.72, 34.06 and 28.88 g/g sorption capacity for diesel, petrol, kerosene and petroleum ether, respectively (Fig. 10). In oil–water medium, decrease in sorption capacities was more pronounced for Bz-GSPF (0.95 g/g for diesel, 1.32 g/g for petrol, 1.46 g/g for kerosene and 1.48 g/g for petroleum ether) as compared to Bz-SPF (0.84 g/g for diesel, 0.96 g/g for petrol, 1.12 g/g for kerosene and 1.11 g/g for petroleum ether). This could be explained on the basis of high hydrophobic character of grafted sorbent as compared to ungrafted one due to presence of hydrophobic poly MMA chains which made it better sorbent for removal of oil in pure oil–medium. Oil capacities of Bz-SPF and Bz-GSPF were found to depend upon time. Initially, up to 30 min rapid sorption was observed, after that there was slow sorption

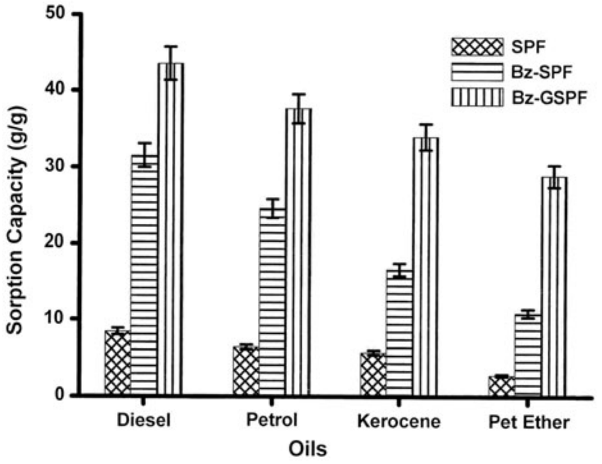

Fig. 9. Sorption capacity of sorbents for different oils in pure oil medium.

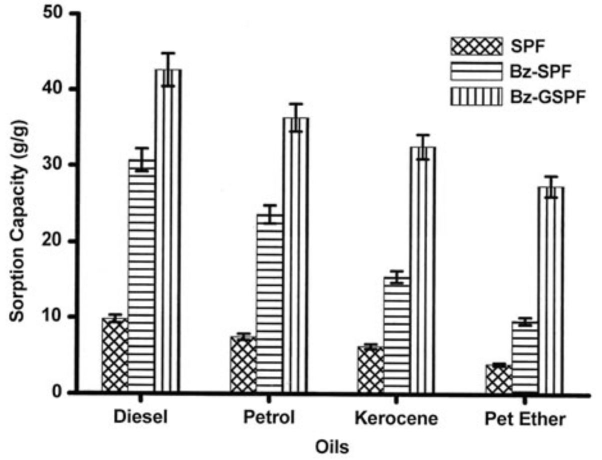

Fig. 10. Sorption capacity of sorbents for different oils in oil–water medium.

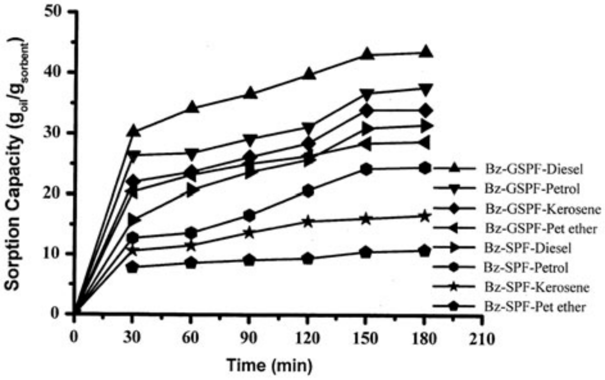

Fig. 11. Effect of time on sorption capacity of Bz-SPF and Bz-GSPF.

in time period of 30–150 min. After 150 min, increase in sorption capacity was almost negligible (Fig. 11). In the initial time up to 30 min fast sorption occurred due to negligible water sorption and surface was available for oil sorption. With time, surface gets exposed to water–oil interface and resulted in low sorption which reached to equilibrium at the end. After 150 min, sorbent gets equilibrated with oil–water interface and showed negligible increase in sorption capacity.

4. Conclusions

The results of this study show that hydrophobicity of SPF was greatly enhanced by benzoylation and graft copolymerization. Response surface methodology studied five process variables in just 31 experimental runs. Perturbation plot indicated KPS:FAS ratio as most significant variable. Monomer concentration of 5 mmol/L with highest range of reaction time (260 min) and lowest range of KPS(1):FAS(1.5) ratio were used to achieve maximum graft yield of 396%. SEM images revealed that surface of fibre become rough on modification, which enhanced the adsorption of oil on fibre surface. Sorption capacities of grafted fibre were more as compared to ungrafted one in both pure oil medium as well as oil–water medium. Higher sorption capacities were observed in pure oil medium as compared to oil–water medium. More importantly, it has been observed that sorption capacities of modified SPF were much higher than those of synthetic sorbents like polypropylene fibre. Thus, SPF is an effective and eco-friendly sorbent for removal of diesel, petrol, kerosene and petroleum ether from water.

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