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# 以膜分离为主的物化法对城市污水中污染因子的去除 特性分析

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摘要:利用高效液相色谱法、三维荧光光谱法分析,考察以膜分离为主的物化处理方式对城市污水中各污染因子的去除特性,尤其对溶解性有机物的富集特性.结果表明,经以膜分离为主的物化方式处理后的城市生活污水属于低碳氮比废水;微絮凝过滤主要富集的是原水中非溶解性有机物和相对分子质量大于30000的芳香类蛋白物质,富集程度在60.93%,尤其是类酪氨酸和类色氨酸;0.45 μm 和0.22 μm 醋酸纤维膜能将相对分子质量大于30000的溶解性有机物全部富集,但是微滤膜对有机物的富集没有选择性;纳滤主要对城市污水中的富里酸类物质和腐殖酸类物质有很好富集效果,富集程度分别为52.01%和53.57%,溶解性有机物的总体富集程度在42%左右.

关键词:城市污水; 膜分离; 能量回收; 有机物富集; 三维荧光光谱

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## Assessing Performance of Pollutant Removal from Municipal Wastewater by Physical and Chemical Methods Based on Membranes

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Abstract: The removal characteristics of various pollution factors in municipal wastewater by membrane-based physical and chemical treatment, especially the enrichment characteristics of dissolved organic matter, were investigated by high performance liquid chromatography and three-dimensional fluorescence spectroscopy. The results showed that the municipal wastewater had a low COD/TN ratio after the treatment. The micro-flocculation filtration mainly enriched the non-dissolvable organic matter in the raw water and aromatic protein substances with relative molecular mass greater than 30 000. The enrichment degree was up to 60.93%, especially for tyrosine-like and tryptophan-like substances. The dissolved organic matter (DOM) with a molecular weight greater than 30 000 was enriched fully by 0.45  $\mu$ m and 0.22  $\mu$ m acetate membranes, but the microfiltration membrane was not selective for the enrichment of organic matter. Thus, NF mainly enriched the fulvic acid and humic acid in urban sewage. The enrichment degree was 52.01% and 53.57%, respectively, and the total enrichment degree of dissolved organic matter was about 42%.

**Key words:** municipal wastewater; membrane separation; energy recycling; organic matter enrichment; three-dimensional fluorescence spectroscopy

2008 年荷兰制定"NEWs 框架",系统地提出了如何回用污水,以及如何回收污水中的能源资源,被称为新一代的污水处理理念<sup>[1]</sup>. 随着能源资源短缺问题日趋严重,越来越多的污水厂选择转型. 典型的案例有荷兰 Dokhaven 厂、新加坡 NEWater 项目和美国得州 EI Paso 项目等<sup>[2~4]</sup>. 无论"NEWs 框架"还是各实践项目,膜技术都因其对原水水量水质的适应性较强、运行和出水稳定、操作简单、节约占地面积等优点而备受推崇<sup>[5,6]</sup>. 在能量回收方面,通常用微滤将非溶解性有机物和溶解性有机物区分开来,分别回收;在污水回用方面,则常用纳滤和反渗透来满足不同的回用要求<sup>[7~12]</sup>.

反观现有的城市污水处理厂却仅旨在污水的达标排放,并未对其中的资源能源以及污水本身进行回收回用.此外,绝大部分城市污水处理厂的处理工艺是以生物处理为主的,但处理的废水却不都是百分百的生活污水.一部分工业废水的汇入使其存在

较高的环境风险性,像污泥上浮、污泥膨胀等问题常有报道<sup>[13,14]</sup>.因此,有必要寻找一种简单、高效且稳定的方法来应急,甚至于作为新一代污水处理厂的工艺参考;回看膜技术在污水处理领域的应用,虽然在能量回收和污水回用方面都发挥着重要的作用,但却忽视了两个问题:一是城市污水中的溶解性有机物成分复杂,不同种类的有机物往往表现出不同的物化特性,而不同级配的膜对有机物的截留特性亦存在差异性,故其回收方式应多元化.二是经膜处理后的废水可作再生水回用,但被膜富集的目标污染物,尤其是溶解性有机物并没有加以回收利用.因此,有必要了解不同级配的膜在污水处理中对溶解性有机物的筛分特性.

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本文选择城市污水厂沉砂池出水为研究对象, 以膜逐级筛分为主的物化法为处理工艺,探究其处 理城市污水的可行性,同时分析不同级配的膜对城 市污水中溶解性有机物的筛分特性,以期为概念污 水厂因地制宜的实践提供多元参考,并为更好地回 收废水中的能量载体——有机物提供理论基础,做 到分类收集,以利于能源转化最大化.

#### 1 材料与方法

#### 1.1 实验流程

为避免无机颗粒对实验结果的影响,取污水厂沉砂池出水为实验原始水样,其基本水质情况见表 1. 首先设计微絮凝过滤单元,选择工业上常用的精制硫酸铝为絮凝剂,目的在于降低原水的 SS,以减轻膜污染;随后设计膜逐级抽滤单元,选择常用的 0. 45、0. 22 和 0. 15 μm 的醋酸纤维膜以及纳滤膜,

探讨不同级配的膜对污水中各类污染物的去除特性,重点探讨膜逐级抽滤对城市污水中有机物的筛分特性.

#### 1.2 实验方法

取上述流程中的各级出水,测其各项常规指标判断以膜逐级筛分为主的物化法处理城市污水的可行性.为减小因检测分析方法带来的实验误差,膜对溶解性有机物的去除特性分析方法如下:首先对实验原始水样中溶解性有机物的相对分子质量及物质含量进行分析,了解其大致相对分子质量范围和各区间的占比情况,以及物质类别和各类别的占比情况.然后对各处理单元不同流程的出水进行采样,分析每个出水水样中溶解性有机物的相对分子质量范围和各区间的占比情况,以及物质类别和各类别占比.最后将每个水样与上一级处理水样进行对比,即可得出每一级处理过程对污水中溶解性有机物的去除特性.

表 1 实验原始水样水质特征

Table 1	Ouality o	of samples	from a	municipal	wastewater	treatment	plant	
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项目	COD/mg·L <sup>-1</sup>	TN/mg·L <sup>-1</sup>	TP/mg·L <sup>-1</sup>	SS/mg·L <sup>-1</sup> NH <sub>4</sub> <sup>+</sup> -N/mg	·L-1 pH
参数	287. 60	53. 63	4. 29	177. 03 29. 62	7. 20

上述方法中,需要说明两点:一是通常人们以能否通过 0.45 μm 膜来区分溶解性有机物和非溶解性有机物,故在本实验中所有膜过滤的出水水样均认为只含有溶解性有机物,且为避免醋酸纤维膜表面的含碳物质对 DOC 测量值的贡献,膜出水的前30 mL水样全部废弃. 二是原水与微絮凝过滤出水的水样须经 GF/F 膜过滤后,方可测量分析.且 GF/F 膜须在 450℃的高温下经马弗炉烘 4 h 后,冷却至常温使用.

#### 1.3 分析方法

#### 1.3.1 水常规六项

COD:重铬酸盐法(GB 11914-89); TP:过硫酸钾消解钼酸铵分光光度法(GB 11893-89); TN:碱性过硫酸钾消解紫外分光光度法(GB 11894-89); NH<sub>4</sub><sup>+</sup>-N:纳氏试剂分光光度法(HJ 5352009); SS:英国 partech740 污泥浓度计; pH:雷磁 phs-3e 计; DOC: Multi 3100N/C 测定仪.

#### 1.3.2 溶解性有机物相对分子质量的测定

采用高效液相色谱测定水样中的相对分子质量,原理是利用已知相对分子质量的标准物质,在一定的实验条件下测得相对分子质量与出峰时间的关系,在相同的实验条件下,根据此关系可由出峰时间反推被测物的相对分子质量<sup>[15]</sup>.色谱柱型号: PL1149-6820 aquagel-OH 20 8 μm 300 × 7.5 mm Agilent,检测器为紫外-可见检测器,设置检测波长

为 254 nm. 流动相为磷酸盐缓冲溶液,流量设置为  $0.5 \text{ mL·L}^{-1}$ ,进样量设置为  $20 \text{ }\mu\text{L}$ . 本实验中选用聚 苯乙烯磺酸钠 (PSS) 为标准物质 ( $M_r$ : 77 000、17 000、6 800、4 300),利用丙酮 ( $M_r$ :58)来校正溶解性有机物的相对分子质量 [ $^{16,17}$ ]. 拟合的曲线如式 ( $^{1}$ ),  $^{2}$  为  $^{2}$  为  $^{2}$  9  $^{2}$  5  $^{2}$  5  $^{2}$  5  $^{2}$  5  $^{2}$  5  $^{2}$  6  $^{2}$  5  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  6  $^{2}$  7  $^$ 

 $\lg M_r = -0.1003 \times t + 4.9957$  (1) 式中,  $M_r$  为相对分子质量; t 为出峰时间, min.

#### 1.3.3 三维荧光光谱

采用 5J1-0004 型荧光分光光度计对样品中的溶解性有机物进行分类、分析. 设置激发波长( $E_x$ ) 200~450 nm,发射波长( $E_m$ ) 范围 250~600 nm. 扫描间隔采用  $E_x$  为 5 nm,  $E_m$  为 5 nm,扫描速度为 2 400 nm·min<sup>-1</sup>,以超纯水作空白样. 测得数据用 Matlab 软件校准后采用寻峰法和荧光区域积分法分析 $\Gamma^{[18-20]}$ .

#### 2 结果与讨论

#### 2.1 常规因子去除特性分析

各级出水的各项常规因子的平均指标如表 2 所示. 分析表格中的数据可以发现,微絮凝过滤对COD、TP和 SS的去除效果极佳,去除率分别为44.86%、89.24%和95.65%. 经 0.45 μm 的醋酸纤维膜过滤后,各项指标均有所下降,其中 COD、TN 和 TP 去除效果较为显著,去除率分别为

本实验各级出水水质料	

Table 2 Quality of samples from every level of efflu
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指标	COD /mg·L <sup>-1</sup>	DOC /mg·L <sup>-1</sup>	TN /mg·L <sup>-1</sup>	TP /mg·L <sup>-1</sup>	SS /mg·L <sup>-1</sup>	NH <sub>4</sub> <sup>+</sup> -N /mg•L <sup>-1</sup>	рН
原水	260. 58	115	67. 375	5. 39	1 840	27. 89	7. 20
微絮凝过滤	143. 68	95	63. 125	0. 58	80	25. 34	7. 10
0.45 μm 过滤	92. 01	75	55. 75	0.06	40	25. 12	7. 21
0. 22 μm 过滤	88. 85	75	53. 875	0.05	0	24. 90	7. 37
0.15 μm 过滤	79. 94	70	50. 25	0. 03	0	24. 01	7. 44
纳滤	55. 78	40	46. 25	0.02	0	18. 55	7. 74

35.96%、11.68%和89.66%.通过对比发现,三级微滤膜除第一级对各项污染因子的去除效果显著外,紧接着的两极微滤膜无论孔径再小,其去除效果较第一级相比甚微.经纳滤的出水,各项污染因子均有明显下降,去除效果显著的指标有COD、TN和NH<sub>4</sub>\*-N,去除率分别为30.22%、7.96%和22.74%.纵观以膜筛分为主的整个物化流程,可发现该物化系统对COD、TP、SS和pH的处理效果极佳,随着原始水样COD的波动,整套物理系统处理效果较为稳定,整体去除率保持在78.59%.但对TN和NH<sub>4</sub>\*-N的去除效果一般,如图1所示.综上所述,经以膜筛分为主的物化法处理后的城市污水为低碳氮比废水,需进一步处理方可达标[21].

# **2.2** 各级膜出水中溶解性有机物相对分子质量分布特征

一定相对分子质量区间内的溶解性有机物往往表现出类似的物化特性<sup>[22]</sup>.城市污水中的有机物成分复杂,在"能量概念工艺中"通常将非溶解性有机物和溶解性有机物区分开来,分别回收<sup>[23]</sup>.溶解性有机物约占城市污水有机物的 30% ~40%,相对分子质量分布范围极广<sup>[24~26]</sup>.按照相对分子质量对不同级配的膜处理城市污水后截留的溶解性有机物进行分类,可以提高对有机物在不同膜处理过程中的规律性认识,同时可以为能量回收和污水回用方法的优化选择提供理论的支持.

本实验采用高效液相色谱法对原水及各级处理 出水中溶解性有机物的相对分子质量进行测定,通 过相应地线性换算,其相对分子质量分布情况如表 3.原水中溶解性有机物相对分子质量分布范围广, 但主要集中在4个水平:大于30000、15000左右、 8500左右和小于7500,其占比分别为3.6809%、 85.8769%、10.1761%和0.2661%.经微絮凝处理 后,DOC下降21%,其中相对分子质量大于30000 的溶解性有机物占比从3.6809%下降至0.8136%, 相对分子质量在1500左右的溶解性有机物占比从 85.8769%下降至67.2045%,说明微絮凝对相对分

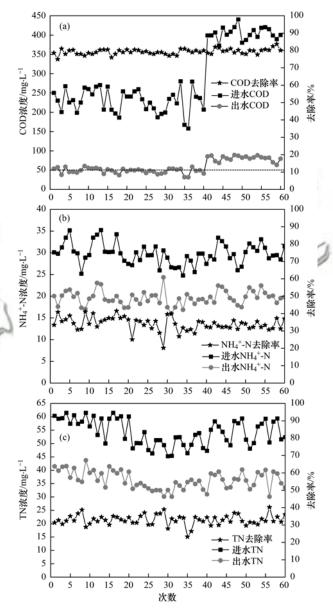


图 1 水质水量波动对 COD、NH<sub>4</sub><sup>+</sup>-N、TN 的去除效果影响

Fig. 1 Effect of fluctuation of water quantity and quality on removal effect of COD, ammonia nitrogen, and total nitrogen removal

子质量大于15 000的大分子溶解性有机物有一定的去除效果. 经 0. 45 μm 和 0. 22 μm 的醋酸纤维膜抽滤后的出水,其色谱图几乎相同. 说明两者对溶解性有机物的去除特性相近,这就是为何许多学者在研究溶解性有机物时会选择 0. 45 μm 和 0. 22 μm 的

微滤膜作前处理. 经 0.45 µm 的醋酸纤维膜抽滤后,DOC 去除率在 6.7%,相对分子质量大于30 000的溶解性有机物已全部去除. 0.15 µm 的醋酸纤维膜和纳滤对溶解性有机物的去除效果显著,DOC 去

除率分别为 14.3%和 41.7%.但所有相对分子质量的占比变化却不明显,说明 0.15 μm 的微滤膜和纳滤对溶解性有机物的去除特性与有机物相对分子质量分布的相关性较小.

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表 3 有机物相对分子质量测定结果

	Table	Results of organic matter me		
项目	出峰时间/min	相对分子质量	所占比例/%	)
	4. 891 0	31 998	3. 680 9	
原水	7. 905 0	15 952	85. 876 9	
<b> </b>	10. 489 0	8 783	10. 176 1	
	11. 175 0	7 496	0. 266 1	
	4. 807 0	32 625	0. 813 6	
	7. 990 0	15 642	67. 204 5	
微絮凝过滤	10. 474 0	8 813	31. 301 3	
	11. 172 0	7 501	0. 379 6	
	11. 653 0	6 712	0. 301 0	
	7. 978 0	15 685	59. 528 8	
	10. 479 0	8 803	39. 340 4	~~
0.45 μm 过滤	11. 123 0	7 586	0. 366 7	011
	11. 656 0	6 708	0. 272 2	14/5
	12. 189 0	5 931	0. 491 9	10
	8. 004 0	15 591	65. 377 2	
	10. 519 0	8 722	33. 346 2	13/1
0. 22 μm 过滤	11. 227 0	7 406	0. 353 3	A1 (0 8
_ / /	11. 653 0	6712	0. 228 1	2/
1 1 /01 1	12. 240 0	5 861	0. 695 2	~
9 811	8. 041 0	15 459	62. 625 4	(
1161	10. 594 0	8 572	34. 896 6	1. 1
0.15 μm 过滤	11. 758 0	6 551	0. 315 1	8
( P/1 V C	13. 518 0	4 363	0. 826 8	
VA VIOLE	19. 234 0	1 165	1. 336 1	
1000	8. 086 0	15 299	66. 974 2	
4	10. 720 0	8 326	29. 819 6	
纳滤	11. 920 0	6 311	0. 565 5	
U-Mari	13. 626 0	4 256	0. 269 9	
	19. 414 0	1 118	2. 370 8	

#### 2.3 三维荧光光谱分析

#### 2.3.1 寻峰法

各级出水中溶解性有机物的三维荧光光谱图如图 2,横坐标为发射波长( $E_{\rm m}$ ),纵坐标为激发波长( $E_{\rm x}$ ).根据 Chen 等[27]描述的荧光峰位置与激发波长、发射波长范围的关系,可识别特定的荧光基团信号.从图 2 可以看出,原水的三维荧光光谱中有一个明显的荧光峰群和一个单峰(峰群 B 和峰  $T_{\rm l}$ ).峰群 B 位于荧光区域  $\mathbb{I}$ ,其荧光峰的中心位于  $E_{\rm x}/E_{\rm m}$  = 275 ~ 280 nm/305 ~ 345 nm,包含类酪氨酸荧光峰和类色氨酸荧光峰.单峰  $T_{\rm l}$  位于荧光区域  $\mathbb{I}$  ,其荧光峰的中心位于  $E_{\rm x}/E_{\rm m}$  = 225 ~ 235 nm/340 ~ 350 nm,属于类色氨酸荧光峰.上述蛋白物质主要来源于城市居民的排泄物、餐厨废液和洗涤废水等.其中峰群 B 的荧光强度还与微生物的代谢有关[28].由于该污水厂处理废水为百分百的生活污水,原水中

的微生物活性较高,其代谢产物的荧光贡献率较大, 故峰群 B 的荧光强度较高[29]; 经微絮凝处理后,峰 群 B 的面积明显减小,仅剩一个单峰 B<sub>1</sub>,其荧光峰 的中心位于  $E_x/E_m = 230 \sim 235 \text{ nm}/340 \sim 350 \text{ nm}$ . 较 原水相比,区域Ⅱ和Ⅳ内荧光峰的强度也明显减弱, 说明微絮凝过滤对类酪氨酸和类色氨酸这两种蛋白 物质的富集效果显著. 经 0.44 µm 的醋酸纤维膜抽 滤后的出水,其荧光谱图与微絮凝过滤后的出水相 比,单峰 B<sub>1</sub> 的荧光面积略有减少,荧光强度降低明 显. 而单峰 T, 并无变化,说明 0.45 µm 的微滤膜对 微生物代谢产生的类酪氨酸有一定的去除效果,但 对污水中的类色氨酸去除效果甚微. 经 0.22 µm 与 0.15 μm 微滤膜处理后的出水,其荧光光谱图与 0.45 μm 微滤膜出水的荧光光谱图差异甚微, 荧光 峰 B, 与荧光峰 T, 仅在荧光强度上略微减弱. 纳滤 出水的荧光光谱图与上级出水相比,其荧光峰的强

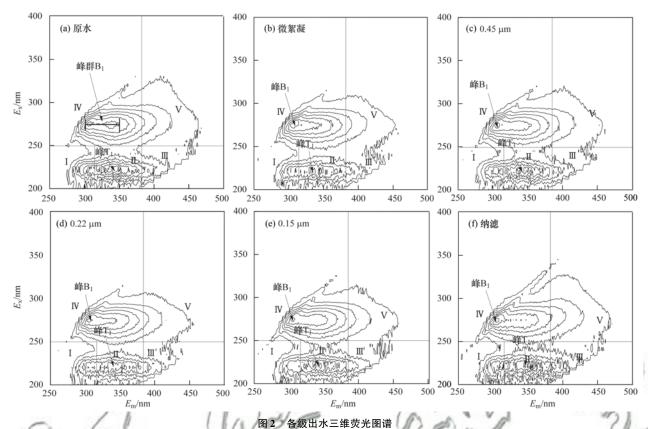


Fig. 2 EEM spectra of every level of effluent

度及面积均有明显变化. 说明纳滤对污水中的蛋白质有很好的去除效果.

#### 2.3.2 荧光区域积分法

荧光区域积分法是按照特定的激发波长和发射波长范围将三维荧光光谱划分成 5 个区域,参考Chen 和姚璐璐等的划分方法 $[30^{-32}]$ :区域 I 代表芳香蛋白类物质 I,荧光范围为  $E_x/E_m=200\sim250$  nm/260~320 nm;区域 II 代表芳香蛋白类物质 II,荧光范围为  $E_x/E_m=200\sim250$  nm/320~380 nm;区域 III 代表富里酸类物质,荧光范围为  $E_x/E_m=200\sim250$  nm/380~550 nm;区域 IV 代表溶解性微生物代谢产物,荧光范围为  $E_x/E_m=250\sim450$  nm/260~380 nm;区域 V 代表腐殖酸类物质,荧光范围为  $E_x/E_m=250\sim450$  nm/380~550 nm.利用 Origin 9.0 软件对各个区域进行积分,然后将所得各区域的积分体积标准化,以反映各区域中特定结构有机物的相对含量[33].

将本实验各级出水中溶解性有机物的三维荧光 光谱图按上述要求积分,得到各区域的标准化积分 后计算各项占比,绘制各级出水荧光区域占比图,即 图 3,横坐标为各级出水,纵坐标  $P_{i,n}$ 为各个区域的 标准化积分体积与所有区域标准化积分体积的比 值. 从中可明显发现,在该物化处理系统中,特定结 构有机物的相对含量仅在微絮凝过滤和纳滤两个处 理单元中出现变化,其余各个单元均无变化,而各级 出水水样的 DOC 含量却都有所降低. 这说明微絮凝 过滤和纳滤对城市污水中溶解性有机物的去除具有 选择性,其选择性与有机物的结构相关. 而孔径为 0.45、0.22 和 0.15 μm 的微滤膜对城市污水中溶解性有机物的去除无选择性.

原水中芳香蛋白类物质 I 的占比为 30.21%, 芳香蛋白类物质 Ⅱ的占比为 18.89%, 富里酸类物 质的占比为18.89%,溶解性微生物代谢产物的占 比为11.33%,腐殖酸类物质占比为20.68%.经微 絮凝过滤后,其出水中五类溶解性有机物的占比依 次变为 14.28%、14.28%、23.81%、23.01%、 24.62%, DOC 浓度也从 115 mg·L<sup>-1</sup>降低至 95 mg·L-1. 从 DOC 及各项占比变化来看,微絮凝过滤 主要对城市污水溶解性有机物中的芳香蛋白类物质 有很好的富集效果,经计算微絮凝过滤对芳香蛋白 类物质 I 的富集程度为 60.93%, 对芳香蛋白类物 质 II 的富集程度为 37.52%. 经 0.15 μm 微滤膜过 滤后的出水中各项占比情况与微絮凝过滤一致,依 次分别为 14.28%、14.28%、23.81%、23.01%、 24.62%,而纳滤后的出水中各项占比均变为 20.00%, DOC 浓度从 70 mg·L<sup>-1</sup>降低至 40 mg·L<sup>-1</sup>. 从 DOC 及各项占比变化来看,纳滤主要对城市污水 溶解性有机物中的富里酸类物质和腐殖酸类物质有

很好的富集效果,经计算纳滤对富里酸类物质的富集程度为 52.01%,对腐殖酸类物质的富集程度为 53.57%.此外,经 0.45 μm 和 0.22 μm 的微滤膜过滤后,城市污水中溶解性有机物的各项占比一致,且所测 DOC 含量相同,故可认为两者对有机物的去除效果相似.这也解释了为何许多研究中为避免非溶解性有机物和无机颗粒对实验的影响,选择 0.45 μm 或 0.22 μm 的微滤膜作前处理<sup>[34]</sup>,以更准确地分析水样中的溶解性有机物.



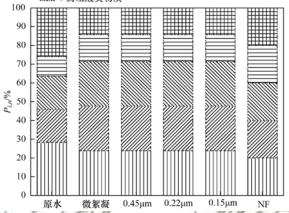


图 3 各级出水荧光区域积分占比
Fig. 3 Proportion of fluorescence regional integration

#### 3 结论

- (1) 经膜逐级筛分为主的物化方式处理后的城市污水属于低碳氮比的废水.
- (2)微絮凝过滤主要富集的是原水中非溶解性 有机物和相对分子质量大于30000的芳香类蛋白物 质,富集程度在60.93%,尤其是类酪氨酸和类色 氨酸.
- (3)0.45 μm 和 0.22 μm 醋酸纤维膜能将相对 分子质量大于30 000的溶解性有机物全部富集,但 是不同孔径微滤膜对有机物的富集没有选择性.
- (4)纳滤主要对城市污水中的富里酸类物质和腐殖酸类物质有很好富集效果,富集程度分别为52.01%和53.57%,溶解性有机物的总体富集程度在42%左右,其富集特性与有机物相对分子质量的相关性较小.

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