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Analysis of infrared optical polishing effluents and reduction of COD and TSS levels by ultrafiltration and coagulation/flocculation

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ABSTRACT

Samples of polishing effluent produced during infrared optics manufacture were analyzed. Their particle size, composition, Zeta potential, chemical oxygen demand (COD), total suspended solids (TSS), and settleable solids were determined. Feasibility of treatment methods such as ultrafiltration (UF) and coagulation/flocculation was investigated to reduce both COD and TSS. It was found that effluents consisted of a suspension of micro- and nanoparticles. Effluent particle size distribution reflected the removal rate of the originating polishing process. Their composition was primarily germanium and other polished substrates as well as polishing abrasives. The effluent Zeta potential was highly negative and prevented particle settling. COD of all specimens was very high, which prevented sewage discharge. Laboratory-scale trials using UF showed substantial COD abatement of up to 74.1%. TSS was reduced to zero after UF. Comparable coagulation/flocculation COD abatement was demonstrated for the highest COD sample.

Keywords: Polishing effluents; Industrial; Wastewater treatment; Analysis; Industry; Effluent

1. Introduction

High value optics manufacture is an established industry in the UK. A wide range of scientific and technological worldwide industries such as medical, defense, and aerospace rely on these precise elements.

Manufacture of high value optics relies heavily on polishing processes. Conventional polishing employs slurries containing abrasive particles that flow between the surface being polished and a pad. The slurry may have a specific pH to generate a slight etching of the surface that in combination with the suspended abrasives removes material from the substrate [1]. Polishing of optical components usually employs a series of progressively finer abrasive media to grind away the higher points of a surface profile until this becomes "smooth." The different polishing stages can be generally classed by the abrasive size employed. For example, larger abrasive particles, of typical average of up to 45 μm, are used for rapid stock removal. Subsequent lapping operations may employ "mid-range" abrasive sizes ranging from 14 to 6 μm. Final polishing is typically achieved using abrasives ranging from 4.5 μm to submicron. Common slurry abrasives include diamond, silicon carbide, alumina, and silica. Polishing slurries can be both oil soluble or emulsions.

Although polishing slurries can be recirculated and are commonly reused, there is a critical point where their abrasive concentration falls below

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optimum. At this point, polishing efficiency is severely affected [2]. Consequently, these "worn" slurries are disposed and a "fresh" batch is prepared. Due to the industry growth [3] and diversification [4,5], effluent volume from optical polishing increases and it needs to be disposed responsibly.

This study focuses on the polishing effluent generated by the infrared (IR) optical industry. This forms a substantial portion of the UK's high value optics industry. The substrate materials typically polished are germanium, silicon, gallium arsenide, zinc selenide, zinc sulphide, glasses, and other miscellaneous IR materials. Night vision systems are the most common applications of these materials. Approximately, 10,000 l of wastewater are generated per year at this facility by 10 dedicated machines included in this study.

Currently, there are a large number of publications related to disposal management of chemical–mechanical polishing (CMP) effluents from the semiconductor industry. Effluent disposal in the photovoltaic industry has also registered a growing interest [6–9]. However, literature relating to the physical or chemical properties, composition, and disposal of IR polishing effluents is still scarce. In this work, we set to determine the composition, particle size distribution, settleable solids (SetSol), chemical oxygen demand (COD), total suspended solids (TSS), and Zeta potential of optical IR polishing effluents from "worn" slurry samples collected from a major European facility. To simplify this analysis, the scope of the research has been on reduction of COD and TSS within local sewage regulations. Ultrafiltration (UF) and coagulation/flocculation have been initially investigated as routes to achieve this. In England and Wales, sewage effluent disposal must conform to the Water Industry Act 1991 [10]. Local consent limits issued for these effluents are of 1,000 and 500 mg/l for COD and TSS, respectively. Other elements detailed in the consent are copper, zinc, cadmium, lead, chromium, and iron. All of these are limited to 2 mg/l.

2. Experimental

Six different waste slurry samples, A–F, were collected from a large UK IR specialist optical polishing facility. The samples were collected at the end of six different production lines. These polishing processes ranged from rapid material removal to final polishing for a range of different substrate materials. Table 1 illustrates some of the sample characteristics noted at an initial visual assessment following their collection. Table 1 also gives the polishing processes originating the samples and abrasive type used.

Characterization of the effluent composition, particle size distribution, COD, SetSol, TSS, and Zeta potential enabled the specification of feasible routes for treatment. Prior to characterization, each sample was thoroughly homogenized by shaking it for 30 s before a subsample was taken. This procedure was determined by initial microscopy homogenization studies.

2.1. Particle size distribution and composition of polishing slurry effluents

Characterization of the slurry particle size distribution is critical for the effective treatment of polishing slurries. This is even more important if treatment techniques such as UF are to be considered. In the present work, particle size distribution was performed using dynamic light scattering (DLS) techniques [11]. This was conducted in two stages to ensure best resolution. Initially, a Mastersizer 2000 (Malvern Instruments, Worcester, UK) was used to characterize the sample solids down to 1 μm. Median and quantiles were calculated. After this, the particle distributions were further characterized using a Nanosizer (ZS Malvern Instruments, Worcester, UK). Prior to this, the samples were filtered to remove all particles over 1 μm. Each sample was characterized at least three times to ensure repeatability. In addition, optical and scanning electron microscopy (SEM) were used to complement the DLS characterization.

The slurry samples were also analyzed to accurately establish their chemical composition. This was conducted using both energy dispersive analyses (EDS) and X-Ray diffraction (XRD). Slurry dry powder specimens were prepared. Typically, 140 ml subsamples of each of the slurries were set in an oven at 105˚C until all fluid had evaporated leaving a dry powder. These analyses also identified unexpected or hazardous residual elements that required special treatment.

2.2. Characterization of polishing effluent COD, SetSol, TSS, and Zeta potential

COD is a measure of the amount of oxygen used in chemical oxidation of inorganic and organic matter contained in wastewater. COD together with other wastewater parameters such as TSS is widely considered an indicator of pollution degree and environmental impact. In order to comply with effluent discharge regulations, wastewater effluents often require treatment to abate COD and TSS prior to sewer disposal.

Table 1
Polishing effluent samples: visual assessment at collection, process origin, and abrasive type used

Sample	Effluent color	Visual assessment comments	Substrate (s) polished	Abrasive slurry type	Typical slurry components (datasheets)	Process
A	Black-gray	• Phase separation present (thin yellow skin) • Black particles in the lower phase	Mainly Ge	Water-diluted fine diamond abrasive	Diamond powder in aqueous solution	Final polishing
B	Yellow-brown	• Foam present • Black precipitates present	ZnSe, Si, and Ge	Water-based lubricant	Water	Smoothing
C	Translucent white	• Neither foam nor precipitated particles are present	Mainly Ge	Water-diluted coolant with mineral oil + antifoams	Mineral oil, corrosion additives, diethanolamine, and petroleum distillates	Rapid material removal
D	Opaque white	• Foam present • Yellow precipitates present	ZnSe and ZnS	Water-diluted fine diamond abrasive + antifoams	Diamond powder in aqueous solution Mineral oil, and petroleum distillates	Final polishing
E	Turbid white	• Fine black precipitates	Mainly Ge, occasionally Si, ZnSe, and ZnS	Water-based fine diamond abrasive + antifoams	Diamond powder in aqueous solution	Final polishing
F	Dense yellow	• Neither foam nor precipitated particles are present	Ge, ZnSe, ZnS, GaAs, Si, and Glass	Semi-synthetic water-soluble cutting fluid	Fatty acid soaps mineral oil, amides, urea, glycerol, and tall oil fatty acids	Lapping

COD values were calculated for all samples using a standard procedure [12] for the examination of water and wastewater. For this, the test effluent is heated up to 148˚C for 2 h with potassium dichromate ($K_2Cr_2O_7$) and concentrated sulphuric acid (H_2SO_4) in the presence of silver (Ag) as catalyst. After cooling, the COD values were measured by photometry (NOVA 60 by MERCK, Darmstadt, Germany). Results were expressed in mg/l.

SetSol were measured using an Imhoff cone in accordance with standard procedures [13]. One liter of each of the samples after thoroughly mixing was allowed to settle. After 1 h, the volume of solids (ml) that settled at the cone apex was measured.

Following this procedure, the TSS was measured [14] and 100 ml of each effluent sample without SetSol were filtered using a Buchner flask and paper filter with particle retention of 1.2 μm (Whatman type GF/C glass-fiber, Leicester, UK). Following filtration, the paper was dried at 105˚C in an oven for 2 h. The net weight of the retained particles was measured using an analytical balance to determine the TSS of each sample:

$$\mathrm{TSS} = \frac{w_2 - w_1}{V} \tag{1}$$

TSS, total suspended solids in mg/l; w_2, paper filter + dried suspended solids residual weight (mg); w_1, paper filter weight; and V, volume of sample filtered (l).

Zeta potential is the electrical potential that exists at the shear plane of a particle. It describes the electrostatic interactions of particles in a fluid and can be used as a measure of its flocculation state. The Zeta potential was measured with a Zetasizer

(Malvern Instruments, Worcestershire, UK) at ambient temperature.

Each experiment was repeated at least twice for repeatability.

2.3. Ultrafiltration

From a manufacturing point of view, UF provides a simplified approach and automation potential for efficient effluent disposal. Successful treatment of some semiconductor CMP effluents using UF and reverse osmosis (RO) [15] and UF preceded by chemical treatment have been previously reported [6]. UF trials were conducted at laboratory scale. The cross-flow filtration pressurized (N_2) system removed micro- and nano-size particles using a porous membrane. Commercially available membranes (Polyethersulfone Biomax™, PB, MERCK, Darmstadt, Germany) with 67 nm pore size were used. These membranes were chosen based on results from the previous particle size distribution measurement. The pressure was set to 0.7 atm. as specified by the membrane manufacturer. Polishing slurry subsamples of 350 ml were filtrated. All experiments were conducted three times to verify repeatability. Following filtration, the COD of the samples was measured to analyze the effect of the filtration process.

2.4. Coagulation/flocculation

Coagulation/flocculation can be potentially a cost-effective way of disposing IR polishing effluents. Coagulation/flocculation is the separation of fine suspended solids brought about using charge neutralization via a coagulant. Coagulation [16,17] and more recently electrocoagulation [18–20] have been recently employed to investigate turbidity, total organic carbon, and COD abatement in semiconductor effluents. Similar techniques have been applied in the photovoltaic industry [7].

The effectiveness of coagulation/flocculation was demonstrated for sample F. This was chosen because of its high COD (6,645 mg/l) and Z potential (−25.1 mV).

In order to ascertain the coagulation mechanisms of polishing effluents, initial titration tests were conducted. Ferric $Fe_2(SO_4)_3$ was used as the coagulant. $Fe_2(SO_4)_3$ is widely used in wastewater treatment [21] and it was readily available to the researchers. Coagulation tests were undertaken on a multi-jar tester. Samples of polishing slurry effluent were dosed with varying amounts of $Fe_2(SO_4)_3$ relating to different Zeta potentials. Conditions involved rapid mix at 300 rpm for 1 min, a 15 min 30 rpm flocculation stage, and a 15 min settlement period [22].

3. Results and discussion

All polishing slurry samples typically had a cloudy appearance consistent with that of particles in suspension. The samples color varied and this reflected the particular components involved in each of the polishing processes i.e. the substrate material, abrasive particles, and solvents. The foam reported on some of the slurry samples (see Table 1) is attributable to aeration occurring during slurry recirculation in the polishing machine. Antifoams are commonly added to polishing slurries to avoid excessive foaming that sometimes occurs. This was confirmed subsequently for samples C, D, and E at the optical factory shop. These antifoams may also contain oils. However, a separate oily phase was not observed in any of these samples. Therefore, it was assumed that any oils were in emulsion.

3.1. Particle size distribution and composition

Fig. 1 illustrates an example of the particle size distribution of a polishing effluent F. A narrow distribution around a mean of 1.15 μm is observed for this sample. This is consistent with the process origin of this sample being optical lapping. This mean particle size is typical of a process with a very low rate of material removal. In general, the particle size distribution from each of the samples reflected the polishing process in which they were employed. Table 2 shows the cumulative particle size distribution parameters. As seen, the median particle size for polishing effluent ranged from 100 to 1 μm approximately. The processes with greater rates of material removal yielded larger particle sizes and vice versa. Table 2 also illustrates the median and range of the submicron particle size distribution for all samples. For this measurement, the effluent mean particle size ranged from 460 to 180 nm. Fig. 2 shows the submicron particle size distribution of sample F.

Fig. 3 is a SEM image for sample D. A continuous layer of particulate ranging from 2 to 4 μm can be observed. This correlates well with the particle size distribution previously obtained (Table 2).

Fig. 4 shows a summary of the elements found in the EDS analysis conducted for each of the samples. A dominant presence of germanium (Ge) was found. This was expected as Ge was the main substrate polished in most machines when the effluent samples were collected. Other substrates polished in the same

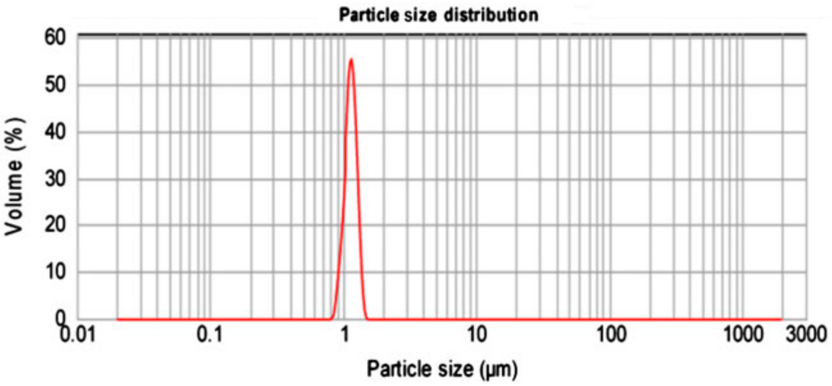

Fig. 1. Particle size distribution obtained using Malvern Mastersizer, sample F.

Table 2
Particle size distribution by sample/process

Sample and process		Particle size characterization parameters (range >1 µm)			Sub-micron characterization parameters (range <1 µm)	
Sample	Process	*d*(0.1) (µm)	*d*(0.5) (µm)	*d*(0.9) (µm)	Mean particle size (nm)	Range (nm)
A	Final polishing	2.35	51.80	480.08	300	932
B	Smoothing	18.73	100.26	205.33	290	922
C	Rapid material removal	2.02	5.39	74.37	460	820
D	Final polishing	2.30	9.27	55.1	180	962
E	Final polishing	1.48	5.57	151.12	290	350
F	Lapping	0.99	1.15	1.294	450	820

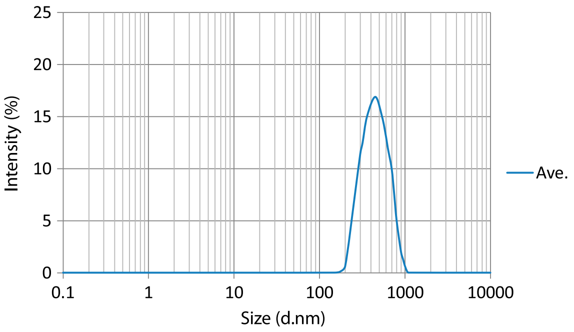

Fig. 2. Submicron particle size distribution, sample E.

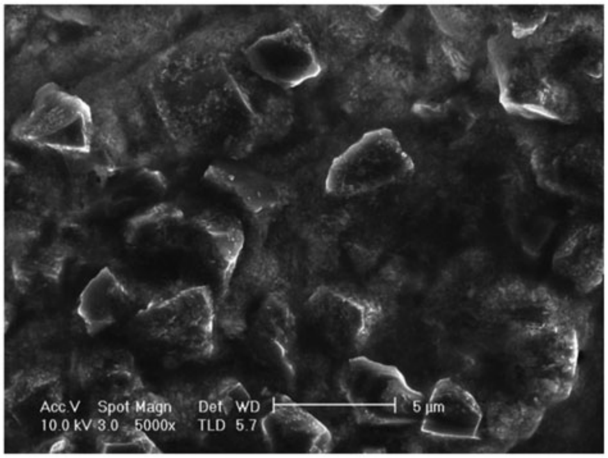

Fig. 3. SEM image for sample D showing effluent particulates.

machines included silicon (Si), zinc selenide (ZnSe), and zinc sulphide (ZnS) and these were also present in the analyses. Further elements found were related to the additives present in the polishing slurry. This was confirmed by their datasheets.

XRD analysis revealed the presence of GeO_2. This was confirmed by SEM microscopy. Fig. 5 shows an image of GeO_2 nanocrystals found as precipitates in sample A. These were most likely related to the abrasives used during the polishing process. Their approximate size is 500 nm. Similar nanocrystals were also found on samples B and E.

In addition to the polished substrates, carbon (C) and oxygen (O) were also present as chemical species in all the results. Samples A and E showed small

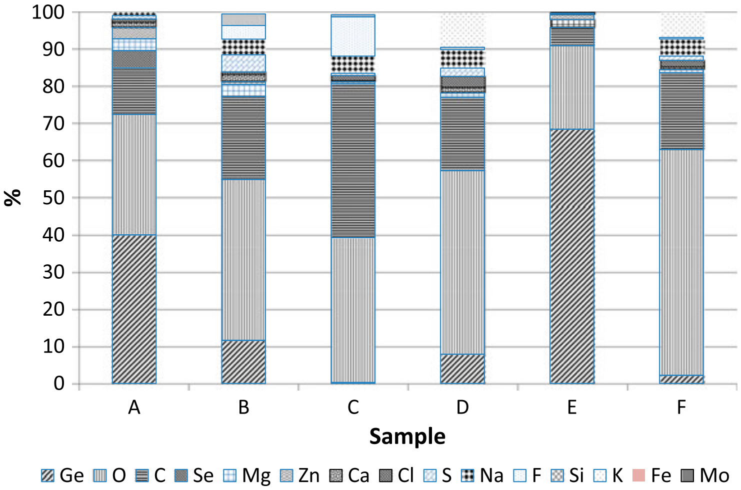

Fig. 4. Summary of elements found by sample.

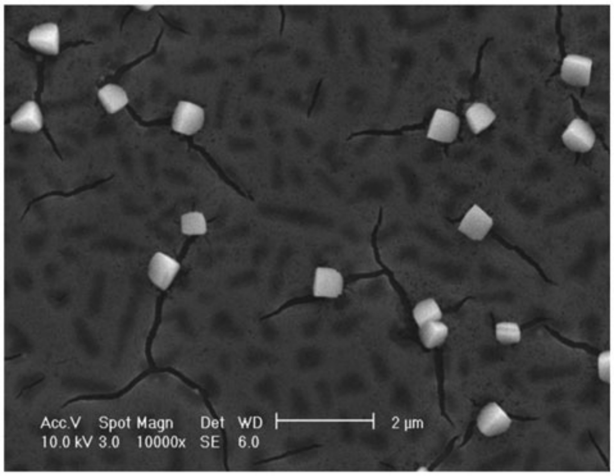

Fig. 5. GeO_2 nanocrystals found in sample A after evaporation.

percentages of up to 4.21% of zinc (Zn) attributable to polished substrate residuals. Iron (Fe) was present in the analysis for sample C in smaller amounts of up to 0.15%. This was not entirely unexpected. Metallic polishing–machine tooling is often made of steel alloys.

In general, the EDS and XRD analysis did not return unexpected elements or compounds. These analyses were clearly dominated by components relating either to the polishing substrates or the slurry abrasive used.

3.2. Polishing effluent COD, SetSol, TSS, and Zeta potential

Table 3
COD, TSS, SetSol, and Zeta potential results summary

Sample	COD (mg/l)	TSS (mg/l)	SetSol (ml/l)	ZPot. (mV)	Oils
A	3,048	341	1.8	−30.2	No
B	4,083	713	0.3	−20.3	No
C	5,688	613	0	−4.8	Yes
D	6,010	4,893	1.1	−37.3	Yes
E	1,789	232	–	−20.3	No
F	6,645	153	0	−25.1	Yes

Table 3 gives a summary of all effluents parameters calculated. The measured COD of all samples as received ranged from 1,789 to 6,645 mg/l. This was much higher than the local consent limit, currently set at 1,000 mg/l.

The measured TSS values of the samples A, E, and F were compliant with the current discharge consent limit at 500 mg/l. Samples B and C showed TSS values were close to this limit, while that of sample D was many times over this limit at 4,893 mg/l. These results correlated well with the particle size distribution measurements previously conducted. The effluents whose distribution overlapped the 1.2 μm filter pore size used had a larger number of particles filtered out and as a result a lower TSS. In contrast, sample D had a smaller submicron particle distribution, and therefore a lower number of particles filtrated out. COD was remeasured following the TSS procedure to observe the effect of filtration. Although 54 and 55% of significant reduction were observed for samples A and B, respectively, the new COD values remained greater than current local sewer discharge consent. The COD reduction observed in effluents A and B was primarily due to their particle size distribution. These samples had the largest particle sizes (see Table 2), with a large number of these above the 1.2 μm filter pore size. In contrast, for samples C–F, a lower COD reduction was observed. The polishing processes in which these effluents originated are the final removal stages in optical finishing. Therefore, a large number of particles in these effluents were in the nm range and this meant that they had a considerable influence in determining the effluent COD. In view of these observations, it was confirmed that UF and coagulation/flocculation were viable treatment routes. UF processes provide enhanced particle retention and an improved chance to abate COD and TSS levels.

Very high negative Z potential values observed contributed to the low SetSol values measured (Table 3). This was consistent with the initial visual assessment which reported that almost or no precipitates were observed. Therefore, employing a settlement tank previous to effluent treatment was not considered advantageous.

Dry preparation of SEM sample led to the discovery of an oil phase in samples C, D, and F. It was subsequently confirmed that oil, antifoams, and other additives had been used on these slurries. As shown in Table 2, the samples with the higher COD had an oil phase. The effect of this oil phase on the samples COD was investigated. Oils were extracted from the polishing effluents and their concentration was determined using the isolute SPE columns procedure [23]. After this, COD was remeasured. The extraction procedure resulted in COD improvements of 11.1, 10.4, and 9.7% for samples C, D and F, respectively. Oil concentration was estimated at 780 and 863 mg/l for samples C and F, respectively. Value for sample D could not be obtained due to column blockage.

3.3. UF trials

UF trials were conducted at laboratory scale using a N_2-pressurized cross-flow filtration system. In view of the particle size distribution obtained, a 64 nm pore size membrane was selected for all effluents. This provided economical means of operation and sufficient level of filtration for most particles. Fig. 6 shows the COD values for each effluent sample before and after UF. As seen, there is a significant improvement for all samples. Samples A, B, C, D, and F showed distinctive COD improvements ranging from 60 to 75%. Sample E, which had the lowest COD at collection (1,789 mg/l), observed a smaller but still favorable 34% abatement. Following UF trials, samples A, B, and E closely approached the current sewage discharge consent COD limit at 1,000 mg/l. We have attributed this to the substantial removal of solids after UF, including some oxidizable, as shown in the EDS and XRD analyses. Further research is necessary to establish its fundamental mechanism.

Following UF, the samples appearance was clean and transparent. Due to the level of filtration used, all particles larger than 1.2 μm were removed. Therefore, the TSS for all samples after UF was of 0 mg/l, within limits set for sewage discharge consent. This was expected. The results were considered an experimental validation of compliance.

A similar treatment strategy was followed by Juang et al. [15]. Polishing effluents from the

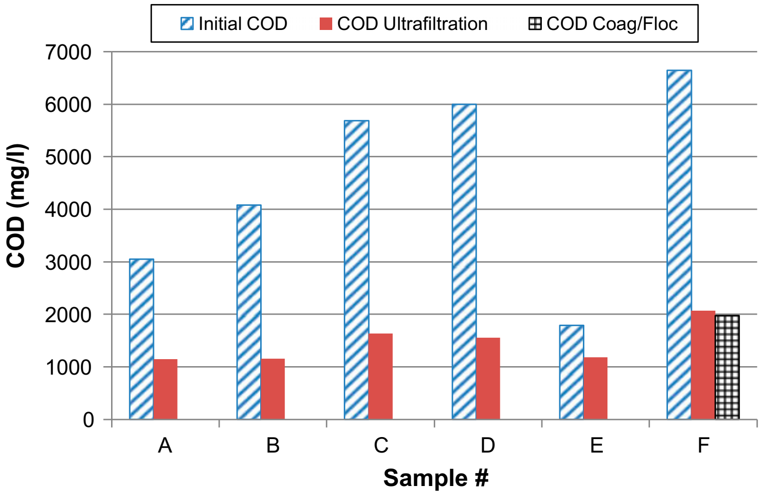

Fig. 6. COD results by sample before and after UF and coagulation/flocculation.

semiconductor industry in Taiwan were treated by filtration first (1 µm), followed by UF (40 nm). Further RO allowed the water permeate to meet tap industrial water reuse standards. For photovoltaic effluents, Drouiche et al. [6] demonstrated the feasibility of UF preceded by chemical pretreatment for the regeneration of cutting fluids.

Laboratory trials have confirmed UF to be an effective method for the treatment of IR effluent, including samples found to have an oily phase (samples C, D, and F). Permeation flux decline caused by fouling and concentration-polarization effects of oils [24–26] and antifoams agents [27] in metal working fluids have been previously reported. Clearly, these effects need to be specifically investigated for IR effluents prior to widespread application of UF processes.

3.4. Coagulation/flocculation

Prior to coagulation/flocculation, titration experiments were conducted to investigate the relation between the amount of coagulant solution (ferric $Fe_2(SO_4)_3$) and the resulting Zeta potential of sample F. Fig. 7 shows the titration curve obtained. Zeta potential (mV) was plotted in relation to the volume of $Fe_2(SO_4)_3$ solution (µl) used. It was observed that when the coagulant solution volume exceeded 14 µl, larger positive Z potentials of over 10 mV were obtained. Conversely, when less than 4 µl were used, the Zeta potential was largely negative and approached those of the initial sample measurements. Based on the observed behavior, a multi-jar test was conducted. Four beakers were used with varying amounts of coagulant corresponding to Z potentials of −10, −5, 0, and 5 mV. SetSol, TSS, and COD were remeasured after coagulation/flocculation. The jar test results are shown in Table 4. As seen, when a Zeta potential of +5 mV was achieved resulted in the highest SetSol, the lowest TSS, and COD. This was close to the optimum range of the Zeta potential previously observed between −11 and +3 mV using this coagulant for raw water [28]. Also, at +5 mV, the SetSol value improved from the initial 0 to 367 ml/l, with a large amount of particles settling and the sample losing its turbidness. The initial TSS value of sample F was already below legal discharge consent as received (153 mg/l); however, this was further reduced to 51 mg/l. In total, the COD was reduced up to 68%, from 6,645 to 1,980 mg/l. A very similar reduction was previously obtained using UF, as shown in Fig. 6.

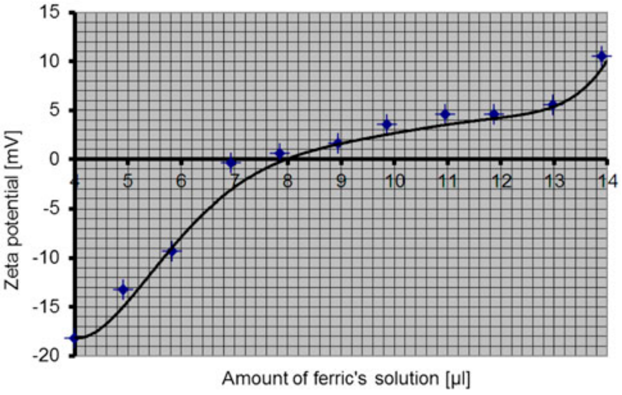

Fig. 7. Titration curve for sample F.

Table 4
Sample F results after coagulation

ZPot. (mV)	Ferric dose (µl)	SetSol (ml/l)	TSS (mg/l)	COD (mg/l)
5	12.8	367	51	1,980
0	8	233	147	2,616
−5	6.5	117	118	3,158
−10	5.6	100	222	3,194

3.5. Discussion summary

When compared, UF and coagulation/flocculation, they both exhibit great potential for the treatment of polishing effluents from the IR industry. COD improvement was virtually the same. UF achieved a further TSS reduction, virtually of 0 mg/l as opposed to the low 51 mg/l obtained with coagulation/flocculation. Nonetheless, this value is well under current discharge consent at 500 mg/l.

Although the COD abatement was substantial, sewage discharge consent level was not achieved by either of the techniques used during this initial work. Further studies should be conducted to optimize both UF and coagulation/flocculation parameters. For example, COD improvement can be potentially achieved by further particle removal via RO, as previously demonstrated by Juang et al. [15]; or chemical pretreatment as demonstrated by Drouiche et al. [6]. Small-scale dilution can also be applied as a simple procedure for samples A, B, and E, since their UF COD was very close to the sewage disposal consent. COD of these will be proportionally reduced to the water volume used for dilution. Evidently the main disadvantage of this is the increase of the volume rate to discharge into the sewer. Nonetheless, The COD and TSS improvements obtained using both UF and coagulation may potentially bring substantial benefits to the environment while leading to cost-effective polishing effluent disposal too. Currently, disposal of these IR polishing effluents is mostly subcontracted and largely relies on techniques such as high volume dilution.

IR optics manufacture involves a number of abrasive polishing processes and a range of substrate materials. It has been found that these produce a range of effluents having different compositions, and physical and chemical properties. As a result, characterization and disposal of the IR effluents cannot be simply generalized.

Significant COD abatement has been achieved by the use of both UF and coagulation/flocculation. In terms of plant operational costs, UF appears to incur the highest costs compared with the simple tank process of electrocoagulation/flocculation. These costs are associated with the supply of pressurized fluids, fouling, membrane replacement, etc. [29].

It is clear from the present study that after the use of UF and coagulation/flocculation, there is no need for large-scale dilution, with reduction in the potential costs incurred and the volume of effluents to be disposed.

4. Conclusions

(1) Particle distribution in optical IR polishing effluents is made of a large number of particles both in the micro and nano range. Measurements performed showed the particles size ranged from a maximum of approximately 480 μm down to a few nm. The particle size distribution was influenced by the polishing process removal rate in which the slurries were used. Higher removal processes yielded larger particle sizes and vice versa.

(2) The chemical composition of optical IR polishing effluents is primarily dominated by the polished substrate. Carbon and oxygen presence was also significant in the analyses. In some of effluents, GeO_2 abrasive nanocrystals were found. Antifoam agents and other polishing slurry additives used in some of the effluents produce residual oils. These had a detrimental effect on their COD.

(3) All samples collected had high COD values that far exceed the local sewage disposal consent. The effluent Z potential was highly negative for most samples. This influenced the low SetSol and the high turbidity observed.

(4) UF is a powerful process for COD abatement in polishing slurries. For all polishing slurries investigated, the COD was substantially reduced. Turbidity in post-UF effluents was removed.

(5) Coagulation/flocculation was demonstrated to be an effective process for COD reduction and is comparable with UF.

(6) TSS were reduced to zero by the use of UF. All particles larger than 1.2 μm were filtered out.

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